Supplementary data for article:

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Supporting information for the paper:

Expanding the scope of In-promoted allylation reaction: 4-(bromomethyl)-1,3-dioxol-2-one as a synthetic equivalent of a 3-arylhydroxyacetone enolate

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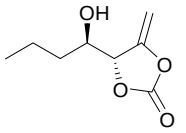
General experimental:

IR spectra were recorded on a Nicolet 6700 FT instrument, and are expressed in cm^{-1}. NMR spectra were recorded on a Varian Gemini 200 (^1H NMR at 200 MHz, ^{13}C NMR at 50 MHz, for samples in deuterated chloroform), and on Bruker Avance III 500 (^1H NMR at 500 MHz, ^{13}C NMR at 125 MHz). Chemical shifts are expressed in ppm (δ) using tetramethylsilane as internal standard, coupling constants (*J*) are in Hz. Reactions induced by microwave irradiation were performed in a Biotage Initiator 2.5. microwave reactor. All chromatographic separations[i] were performed on Silica, 10-18, 60A, ICN Biomedicals. Standard techniques were used for the purification of reagents and solvents.[ii] Mass spectra were obtained on Agilent technologies 6210 TOF LC/MS instrument (LC: series 1200). Microanalyses were performed at the Vario EL III instrument CHNOS Elementar Analyzer, Elementar Analysensysteme GmbH, Hanau-Germany. Melting points were determined on a Kofler hot-stage apparatus and are uncorrected.

Preparation of starting enol carbonates

Enol carbonates **2a-d** were prepared according literature procedures. [iii]

4-(1-Hydroxybutyl)-5-methylene-1,3-dioxolan-2-one ***2e***

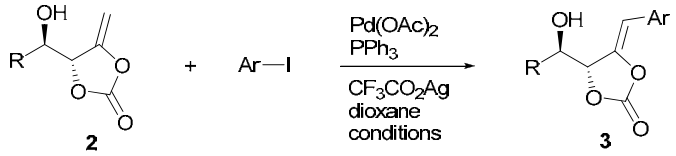


Butyraldehide (27 mg, 33μL, 0.37 mmol) was added to a mixture of 4-(bromomethyl)-1,3-dioxol-2-one **1** (100 mg, 0.56 mmol), indium (64 mg, 0.56mmol), THF (1 mL) and water (2 mL), and the reaction mixture was stirred at rt. The reaction was monitored by TLC (eluent: 20% EtOAc in petroleum-ether) and it was complete after 15 min. The reaction mixture was diluted with dichloromethane (10 mL) and water (10 mL), the aqueous layer was extracted with dichloromethane (2 x 10 mL), combined organic extracts were dried over anh. $MgSO_4$, filtered, concentrated under reduced pressure and the crude product was purified by dry-flash chromatography (SiO_2; eluent: 20% EtOAc in petroleum-ether). 4-(1-Hydroxybutyl)-5-methylene-1,3-dioxolan-2-one was obtained as viscous oil (51 mg, 89 %)

Physical data for **2e:** FT-IR (film, cm^{-1}): 3475, 2963, 2938, 2875, 1830, 1690, 1152, 1059. ^1H NMR ($CDCl_3$, 200 MHz): 5.07-5.02 (m, 1H), 4.97 (dd, J_1 = 3.9 Hz, J_2 = 2.3 Hz, 1H), 4.50 (dd, J_1 = 3.9 Hz, J_2 = 1.7 Hz,, 1H), 3.92-3.79 (m, 1H), 2.62 (d, J = 3.4 Hz, 1H), 1.59-1.34 (m, 4H), 0.97 (t, *J* = 7.0 Hz, 1H). ^{13}C NMR ($CDCl_3$, 50 MHz): 152.4, 149.8,

89.0, 82.1, 71.6, 32.9, 18.6, 13.7. HRMS (ESI): calcd. for [$C_8H_{12}O_4$ + NH_4^+]: 190.1074, found for [M+NH_4]$^+$: 190.1073.

Experimental procedures for the Heck reactions



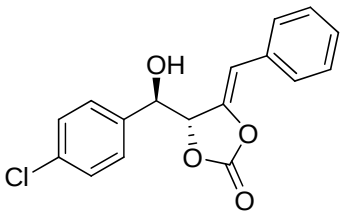
General procedure for the Heck reaction under thermal conditions

To a solution of enol carbonate **2** (0.1 mmol) in dioxane (1 mL) were added aryliodide (0.15 mmol), silver trifluoroacetate (0.15 mmol), palladium acetate (0.02 mmol) and triphenylphosphine (0.02 mmol) under an argon atmosphere. The reaction mixture was vigorously stirred and heated to 95 °C, while the progress of the reaction was monitored by TLC (SiO_2 plates, eluent: 30% EtOAc in benzene). Upon completion, the reaction mixture was partitioned between water and EtOAc, the water layer was extracted with EtOAc and the combined organic extract was dried over anh. $MgSO_4$, filtered and concentrated under reduced pressure. Purification by dry-flash chromatography (SiO_2; eluent: 20% EtOAc in petroleum-ether) afforded of the title compound **3.**

General procedure for the Heck reaction under microwave conditions

A solution of enol carbonate **2** (1.0 mmol) in dioxane (1.5 mL) is put in a microvawe tube, equiped with a magnetic stirring bar and a septum. To this solution were added (in the following order): aryliodide (0.12 mmol), silver trifluoroacetate (0.12 mmol), palladium acetate (7.5 µmol) and triphenylphosphine (7.5 µmol), under an argon atmosphere. A tube with the reaction mixture was transferred into a microwave reactor (Biotage Initiator 2.5) and irradiated with a 160 W, over 30 min. An additional amount of palladium acetate (7.5 µmol) and triphenylphosphine (7.5 µmol) was added and irradiation was continued for additional 20 min, when the reaction was complete. Work-up as for the thermally induced reaction afforded compound **3**.

*4-Benzylidene-5-((4-chlorophenyl)-hydroxymethyl)-1,3-dioxolan-2-one **3a***



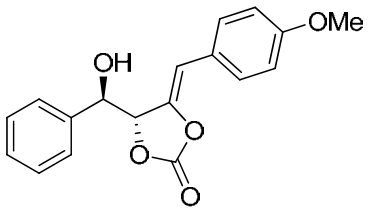
Acording to the general procedure for the Heck reaction under thermal conditions, starting from 4-((4-chlorophenyl)(hydroxy)methyl)-5-methylene-1,3-dioxolan-2-one **2a** (23.5 mg, 0.098 mmol), iodobenzene (30.0 mg, 17µL, 0.15 mmol), silver trifluoroacetate

(33.2 mg, 0.15 mmol), palladium acetate (4.4 mg, 0.02 mmol) and triphenylphosphine (5.1 mg, 0.02 mmol), after purification by dry-flash chromatography (SiO_2; eluent: 20% EtOAc in petroleum-ether), 18.0 mg (58%) of the title compound **3a** was obtained as white crystals.

Acording to the general procedure for the Heck reaction under thermal conditions, starting from 4-((4-chlorophenyl)(hydroxy)methyl)-5-methylene-1,3-dioxolan-2-one **2a** (23.5 mg, 0.098 mmol), iodobenzene (30.0 mg, 17µL, 0.15 mmol), silver trifluoroacetate (33.2 mg, 0.15 mmol), palladium acetate (4.4 mg, 0.02 mmol) and triphenylphosphine (5.1 mg, 0.02 mmol), after purification by dry-flash chromatography (SiO_2; eluent: 20% EtOAc in petroleum-ether), 18.9 mg (61%) of the title compound **3a** was obtained as white crystals.

Physical data for **3a:** mp 115-117 ^{o}C; FT-IR (film, cm^{-1}): 3475, 3029, 1832, 1705, 1494, 1370, 1232, 1129, 1086, 1054, 762, 697. ^{1}H NMR ($CDCl_3$, 500 MHz): 7.40-7.24 (m, 9H), 5.32 (dd, J_1 = 3.7 Hz, J_2 = 1.8 Hz, 1H), 5.13 (d, J = 3.7 Hz, 1H), 5.09 (d, J = 1.5 Hz, 1H), 2.86 (bs, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz): 152.1 (C), 140.6 (C), 134.9 (C), 134.8 (C), 131.9 (C), 128.9 (CH), 128.7 (CH), 128.6 (CH), 128.0 (CH), 127.9 (CH), 106.0 (CH), 82.6 (CH), 73.7 (CH). HRMS (ESI): calcd. for [$C_{17}H_{13}ClO_4$ + NH_4^+]: 334.0841, found for [M+NH_4]$^+$: 334.0839.

4-(Hydroxy(phenyl)methyl)-5-(4-methoxybenzylidene)-1,3-dioxolan-2-one ***3b***



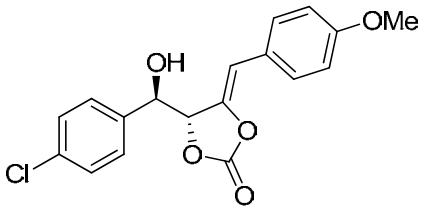
Acording to the general procedure for the Heck reaction under thermal conditions, starting from 4-(hydroxy(phenyl)methyl)-5-methylene-1,3-dioxolan-2-one **2b** (20.0 mg, 0.097 mmol), 4-iodoanisole (31.8 mg, 0.15 mmol), silver trifluoroacetate (32.2 mg, 0.15 mmol), palladium acetate (4.4 mg, 0.019 mmol) and triphenylphosphine (5.0 mg, 0.019 mmol), after purification by dry-flash chromatography (SiO_2; eluent: 30% EtOAc in petroleum-ether), 15.4 mg (51%) of the title compound **3b** was obtained as a yellow oil.

Acording to the general procedure for the Heck reaction under microwaves conditions, starting from 4-(hydroxy(phenyl)methyl)-5-methylene-1,3-dioxolan-2-one **2b** (15.0 mg, 0.073 mmol), 4-iodoanisole (24.0 mg, 0.12 mmol), silver trifluoroacetate (24.2 mg, 0.12 mmol), palladium acetate (3.3 mg, 0.015 mmol) and triphenylphosphine (4.0 mg, 0.015

mmol), after purification by dry-flash chromatography (SiO_2; eluent: 30% EtOAc in petroleum-ether), 11.2 mg (49%) of the title compound **3b** was obtained as a yellow oil.

Physical data for **3b**: FT-IR (film, cm^{-1}):3447, 2932, 1823, 1512, 1251, 1183, 1051, 762, 703, 623. ^1H NMR ($CDCl_3$, 500 MHz): 7.40 (s, 5H), 7.34 (d, *J* = 8.8 Hz, 2H), 6.84 (d, *J* = 8.8 Hz, 2H), 5.35 (dd, J_1 = 3.5 Hz, J_2 = 1.5 Hz, 1H), 5.15 (bs, 1H), 5.01 (d, *J* = 1.5 Hz, 1H), 3.80 (s, 3H), 2.62 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz): 159.1 (C), 152.3 (C), 139.1 (C), 136.5 (C), 130.0 (CH), 128.9 (CH), 128.7 (CH), 126.6 (CH), 124.8 (C), 114.0 (CH), 105.5 (CH), 82.8 (CH), 74.3 (CH), 55.3 (CH_3). HRMS (ESI): calcd. for [$C_{18}H_{16}O_5$ + NH_4^+]: 330.1336, found for [M+NH_4]$^+$: 330.1341.

4-((4-Chlorophenyl)(hydroxy)methyl)-5-(4-methoxybenzylidene)-1,3-dioxolan-2-one ***3c***



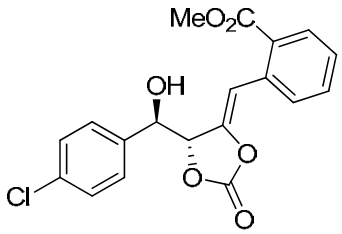
Acording to the general procedure for the Heck reaction under thermal conditions, starting from 4-((4-chlorophenyl)(hydroxy)methyl)-5-methylene-1,3-dioxolan-2-one **2a** (40.0 mg, 0.17 mmol), 4-iodoanisole (56.0 mg, 0.25 mmol), silver trifluoroacetate (56.0 mg, 0.25 mmol), palladium acetate (7.6 mg, 0.034 mmol) and triphenylphosphine (9.0 mg, 0.034 mmol), after purification by dry-flash chromatography (SiO_2; eluent: 20% EtOAc in petroleum-ether), 29.0 mg (55%) of the title compound **3c** was obtained as a yellow oil.

Acording to the general procedure for the Heck reaction under microwave conditions, starting from 4-((4-chlorophenyl)(hydroxy)methyl)-5-methylene-1,3-dioxolan-2-one **2a** (40.0 mg, 0.17 mmol), 4-iodoanisole (56.0 mg, 0.25 mmol), silver trifluoroacetate (56.0 mg, 0.25 mmol), palladium acetate (7.6 mg, 0.034 mmol) and triphenylphosphine (9.0 mg, 0.034 mmol), after purification by dry-flash chromatography (SiO_2; eluent: 20% EtOAc in petroleum-ether), 26.0 mg (50%) of the title compound **3c** was obtained as a yellow oil.

Physical data for **3c**: FT-IR (film, cm^{-1}): 3467, 2932, 1820, 1702, 1512, 1252, 1129, 1080, 857, 764, 739. ^1H NMR ($CDCl_3$, 500 MHz): 7.41-7.34 (m, 6H), 6.86 (d, *J* = 9.0 Hz, 2H), 5.31 (dd, J_1 = 4.0 Hz, J_2 = 1.5 Hz, 1H), 5.11 (bs, 1H), 5.08 (d, *J* = 1.5 Hz, 1H), 3.81 (s, 3H), 2.60 (d, *J* = 4.0 Hz, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz): 159.0 (C), 152.1 (C), 138.9 (C), 135.0 (C), 134.9 (C), 130.1 (CH), 128.9 (CH), 128.0 (CH), 124.6 (C), 114.1 (CH), 105.7 (CH), 82.5 (CH), 73.9 (CH), 55.3 (CH_3). HRMS (ESI): calcd. for [$C_{18}H_{15}ClO_5$

+ NH_4^+]: 364.0946, found for $[M+NH_4]^+$: 364.0945. Microanal: calcd. for $C_{18}H_{15}ClO_5$: C 62.33, H 4.33; found: C 62.01, H 4.42.

Methyl 2-((4-chlorophenyl)(hydroxy)methyl)-2-oxo-1,3-dioxolan-4-ylidene)methyl)benzoate ***3d***

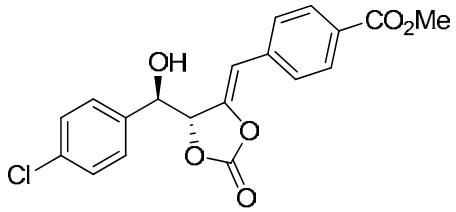


Acording to the general procedure for the Heck reaction under thermal conditions, starting from 4-((4-chlorophenyl)(hydroxy)methyl)-5-methylene-1,3-dioxolan-2-one **2a** (40.0 mg, 0.17 mmol), methyl 2-iodobenzoate (65.0 mg, 0.25 mmol), silver trifluoroacetate (56.3 mg, 0.25 mmol), palladium acetate (8.0 mg, 0.034 mmol) and triphenylphosphine (8.8 mg, 0.034 mmol), after purification by dry-flash chromatography (SiO_2; eluent: 20% EtOAc in petroleum-ether), 31.2 mg (50%) of the title compound **3d** was obtained as a yellow oil.

Acording to the general procedure for the Heck reaction under microwave conditions, starting from 4-((4-chlorophenyl)(hydroxy)methyl)-5-methylene-1,3-dioxolan-2-one **2a** (10.0 mg, 0.042 mmol), methyl 2-iodobenzoate (16.3 mg, 0.063 mmol), silver trifluoroacetate (14.1 mg, 0.63 mmol), palladium acetate (2.0 mg, 0.0084 mmol) and triphenylphosphine (2.2 mg, 0.0084 mmol), after purification by dry-flash chromatography (SiO_2; eluent: 20% EtOAc in petroleum-ether), 6.3 mg (40%) of the title compound **3d** was obtained as a yellow oil.

Physical data for **3d**: FT-IR (film, cm^{-1}): 3479, 2953, 1835, 1720, 1491, 1299, 1269, 1126, 1085, 980, 762. ^{1}H NMR ($CDCl_3$, 500 MHz): 7.96 (dd, J_1 = 7.8 Hz, J_2 = 1.6 Hz, 1H), 7.66 (dd, J_1 = 7.8 Hz, J_2 = 1.6 Hz, 1H), 7.55 (td, J_1 = 7.5 Hz, J_2 = 1.6 Hz, 1H), 7.45-7.35 (m, 5H), 6.23 (d, J = 1.5 Hz, 1H), 5.36 (dd, J_1 = 4.3 Hz, J_2 = 1.5 Hz, 1H), 5.04 (d, J = 4.3 Hz, 1H), 3.88 (s, 3H), 3.44 (bs, 1H). ^{13}C NMR ($CDCl_3$, 500 MHz): 167.6 (C), 151.7 (C), 142.2 (C), 135.3 (C), 134.7 (C), 133.0 (C), 132.5 (CH), 130.7 (CH), 130.4 (CH), 128.9 (CH), 128.3 (C), 128.1 (CH), 127.7 (CH), 104.0 (CH), 82.5 (CH), 74.2 (CH), 52.4 (CH_3). HRMS (ESI): calcd. for [$C_{19}H_{15}ClO_6$ + NH_4^+]: 392.0895, found for $[M+NH_4]^+$: 392.0894.

Methyl 4-((4-chlorophenyl)(hydroxy)methyl)-2-oxo-1,3-dioxolan-4-ylidene)methyl)benzoate ***3e***

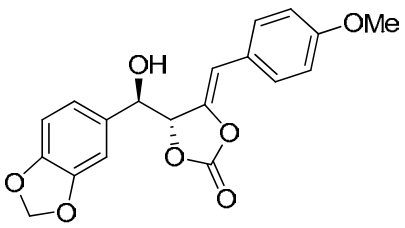


Acording to the general procedure for the Heck reaction under thermal conditions, starting from 4-((4-chlorophenyl)(hydroxy)methyl)-5-methylene-1,3-dioxolan-2-one **2a** (10.0 mg, 0.042 mmol), methyl 4-iodobenzoate (16.0 mg, 0.06 mmol), silver trifluoroacetate (13.3 mg, 0.06 mmol), palladium acetate (2.0 mg, 0.008 mmol) and triphenylphosphine (2.2 mg, 0.008 mmol), after purification by dry-flash chromatography (SiO_2; eluent: 20% EtOAc in petroleum-ether), 9.6 mg (61%) of the title compound **3e** as yellowish crystals.

Acording to the general procedure for the Heck reaction under microwave conditions, starting from 4-((4-chlorophenyl)(hydroxy)methyl)-5-methylene-1,3-dioxolan-2-one **2a** (10.0 mg, 0.042 mmol), methyl 4-iodobenzoate (16.0 mg, 0.06 mmol), silver trifluoroacetate (13.3 mg, 0.06 mmol), palladium acetate (2.0 mg, 0.008 mmol) and triphenylphosphine (2.2 mg, 0.008 mmol), after purification by dry-flash chromatography (SiO_2; eluent: 20% EtOAc in petroleum-ether), 7.2 mg (46%) of the title compound **3e** as yellowish crystals.

Physical data for **3e**: mp 163-164 ^{o}C; FT-IR (KBr, cm^{-1}): 3481, 2955, 2928, 1835, 1722, 1438, 1288, 1188, 1116, 1087, 767. ^{1}H NMR ($CDCl_3$, 200 MHz): 7.97 (d, J = 8.4 Hz, 2H), 7.47 (d, J = 8.4 Hz, 2H), 7.41-7.37 (m, 4H), 5.35 (dd, J_1 = 3.9 Hz, J_2 = 1.6 Hz, 1 H), 5.18 (bs, 1H), 5.12 (d, J = 1.6 Hz, 1H), 3.91 (s, 3H), 2.93 (d, J = 3.9 Hz, 1H). ^{13}C NMR ($CDCl_3$, 50 MHz): 166.7 (C), 142.6 (C), 136.4 (C), 135.2 (C), 134.8 (C), 129.9 (CH), 129.1 (C), 129.0 (CH), 128.5 (CH), 127.9 (CH), 127.5 (C), 105.1 (CH), 82.6 (CH), 73.6 (CH), 52.2 (CH_3). HRMS (ESI): calcd for [$C_{19}H_{15}ClO_6$ + NH_4^+]: 392.0557, found for [M+NH_4]$^+$: 392.0907.

4-(Benzo[d][1,3]dioxol-5-yl(hydroxy)methyl)-5-(4-methoxybenzylidene)-1,3-dioxolan-2-one ***3f***

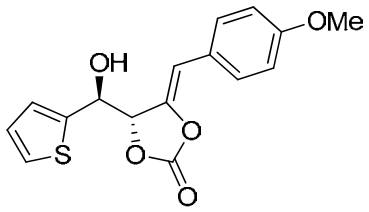


Acording to the general procedure for the Heck reaction under thermal conditions, starting from 4-(benzo[d][1,3]dioxol-5-yl(hydroxy)methyl)-5-methylene-1,3-dioxolan-2-one **2c** (40.0 mg, 0.16 mmol), 4-iodoanisole (57.0 mg, 0.24 mmol), silver trifluoroacetate (53.0 mg, 0.24 mmol), palladium acetate (7.0 mg, 0.032 mmol) and triphenylphosphine (8.4 mg, 0.032 mmol), after purification by dry-flash chromatography (SiO_2; eluent: 20% EtOAc in petroleum-ether), 30.0 mg (53%) of the title compound **3f** was obtained as a yellow oil.

Acording to the general procedure for the Heck reaction under microwave conditions, starting from 4-(benzo[d][1,3]dioxol-5-yl(hydroxy)methyl)-5-methylene-1,3-dioxolan-2-one **2c** (40.0 mg, 0.16 mmol), 4-iodoanisole (57.0 mg, 0.24 mmol), silver trifluoroacetate (53.0 mg, 0.24 mmol), palladium acetate (7.0 mg, 0.032 mmol) and triphenylphosphine (8.4 mg, 0.032 mmol), after purification by dry-flash chromatography (SiO_2; eluent: 20% EtOAc in petroleum-ether), 23.0 mg (40%) of the title compound **3f** was obtained as a yellow oil.

Physical data for 3f: FT-IR (film, cm^{-1}): 3500, 2927, 1827, 1513, 1255, 1130, 1042. ^1H NMR ($CDCl_3$, 500 MHz): 7.38 (d, J = 8.3 Hz, 2H), 6.88 (d, J = 1.8 Hz, 1H), 6.86 (s, 1H), 6.84 (d, J = 1.8 Hz, 1H), 6.81 (d, J = 8.3 Hz, 2H), 5.98 (s, 2H), 5.28 (dd, J_1 = 4.0 Hz, J_2 = 1.9 Hz, 1H), 5.19 (d, J = 1.9 Hz, 1H), 4.99 (bs, 1H), 3.80 (s, 3H), 2.74 (d, J = 3.5 Hz, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz): 159.1 (C), 152.3 (C), 148.0 (C), 147.9 (C), 139.4 (C), 130.5 (C), 130.0 (CH), 124.8 (C), 120.3 (CH), 114.0 (CH), 108.3 (CH), 107.0 (CH), 105.4 (CH), 101.3 (CH_2), 82.8 (CH), 74.2 (CH), 55.3 (CH_3). HRMS (ESI): calcd. for [$C_{19}H_{16}O_7$ + NH_4^+]: 374.1234, found for $[M+NH_4]^+$: 374.1238.

4-(Hydroxy(thiophen-2-yl)methyl)-5-(4-methoxybenzylidene)-1,3-dioxolan-2-one ***3g***

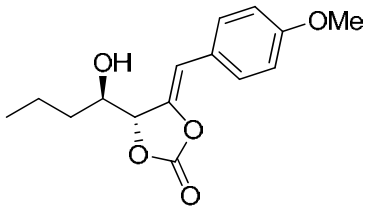


Acording to the general procedure for the Heck reaction under thermal conditions, starting from (4-(hydroxy(thiophen-2-yl)methyl)-5-methylene-1,3-dioxolan-2-one **2d** (27.5 mg, 0.13 mmol), 4-iodoanisole (42.6 mg, 0.2 mmol), silver trifluoroacetate (43.1 mg, 0.2 mmol), palladium acetate (5.8 mg, 0.026 mmol) and triphenylphosphine (6.8 mg, 0.026 mmol), after purification by dry-flash chromatography (SiO_2; eluent: 20% EtOAc in petroleum-ether), 30.0 mg (53%) of the title compound **3g** was obtained as a yellow oil.

Acording to the general procedure for the Heck reaction under microwave conditions, starting from (4-(hydroxy(thiophen-2-yl)methyl)-5-methylene-1,3-dioxolan-2-one **2d** (30.4 mg, 0.14 mmol), 4-iodoanisole (46.0 mg, 0.21 mmol), silver trifluoroacetate (46.0 mg, 0.21 mmol), palladium acetate (6.3 mg, 0.028 mmol) and triphenylphosphine (7.3 mg, 0.028 mmol), after purification by dry-flash chromatography (SiO_2; eluent: 20% EtOAc in petroleum-ether), 21.0 mg (47%) of the title compound **3g** was obtained as a yellow oil.

Physical data for **3g**: FT-IR (film, cm^{-1}): 3463, 2960, 1830, 1705, 1609, 1515, 1254, 1187, 1130, 1085, 858, 712. ^1H NMR ($CDCl_3$, 500 MHz): 7.38 (d, J = 9.0 Hz, 2H), 7.34 (dd, J_1 = 5.0 Hz, J_2 = 1.5 Hz, 1H), 7.09-7.08 (m, 1H), 7.04 (dd, J_1 = 5.0 Hz, J_2 = 4.5 Hz, 1H), 6.84 (d, J = 9.0 Hz, 2H), 5.39 (dd, J_1 = 3.7 Hz, J_2 = 1.7 Hz, 1H), 5.28 (d, J = 3.7 Hz, 1H), 5.26 (d, J = 1.7 Hz, 1H), 3.79 (s, 3H), 3.12 (bs, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz):159.1 (C), 152.2 (C), 139.8 (C), 130.1 (CH), 128.3 (C), 127.2 (CH), 126.1 (CH), 125.5 (CH), 124.7 (C), 114.0 (CH), 105.5 (CH), 82.5 (CH), 71.4 (CH), 55.3 (CH_3). HRMS (ESI): calcd. for [$C_{16}H_{14}SO_5$ + NH_4^+]: 336.0900, found for $[M+NH_4]^+$: 336.0900.

*4-(1-Hydroxybutyl)-5-(4-methoxybenzylidene)-1,3-dioxolan-2-one **3h***



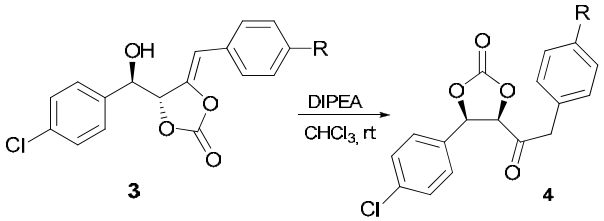
Acording to the general procedure for the Heck reaction under thermal conditions, starting from 4-(1-hydroxybutyl)-5-methylene-1,3-dioxolan-2-one **2e** (30 mg, 0.19 mmol), 4-iodoanisole (66.0 mg, 0.29 mmol), silver trifluoroacetate (66.3 mg, 0.30 mmol), palladium acetate (8.5 mg, 0.038 mmol) and triphenylphosphine (10.0 mg, 0.038 mmol), after purification by dry-flash chromatography (SiO_2; eluent: 20% EtOAc in petroleum-ether), 20.0 mg (38 %) of the title compound **3h** was obtained as a yellow oil.

Acording to the general procedure for the Heck reaction under microwave conditions, starting from 4-(1-hydroxybutyl)-5-methylene-1,3-dioxolan-2-one **2e** (56.0 mg, 0.54 mmol), 4-iodoanisole (118.0 mg, 0.54 mmol), silver trifluoroacetate (119.0 mg, 0.54 mmol), palladium acetate (16.1 mg, 0.072 mmol) and triphenylphosphine (18.8 mg, 0.072 mmol), after purification by dry-flash chromatography (SiO_2; eluent: 20% EtOAc in petroleum-ether), 39.4 mg (40%) of the title compound **3h** was obtained as a yellow oil.

Physical data for **3h**: FT-IR (film, cm^{-1}): 3491, 2963, 2365, 1828, 1706, 1611, 1516, 1254, 1188, 1266, 1053. ^1H NMR ($CDCl_3$, 500 MHz): 7.49 (d, J = 8.8 Hz, 2H), 6.89 (d,

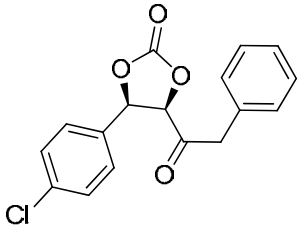
J = 8.8 Hz, 2H), 5.61 (d, *J* = 1.8 Hz, 1H), 5.12 (dd, J_1 = 4.0 Hz, J_2 = 1.8 Hz, 1H), 3.89-3.84 (m, 1H), 3.82 (s, 3H), 2.12 (d, *J* = 7.0 Hz, 1H), 1.61-1.58 (m, 3H), 1.44-1.42 (m, 1H), 0.98 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR ($CDCl_3$, 125 MHz): 159.1 (C), 152.3 (C), 140.4 (C), 130.1 (CH), 124.8 (C), 114.1 (CH), 104.8 (CH), 82.7 (CH), 72.5 (CH), 55.3 (CH_3), 33.3 (CH_2), 18.7 (CH_2), 13.8 (CH_3). HRMS (ESI): calcd. for [$C_{15}H_{18}O_5$ + NH_4^+]: 296.1492, found for [M+NH_4]$^+$: 296.1489.

General procedure for the isomerization of carbonates 3a-c:



A solution of carbonate **3** (0.08 mmol) and DIPEA (0.04 mmol) in chloroform (1.0 mL) was stirred at rt for 1 h. The solvent was removed under reduced pressure and the crude product was purified by dry-flash chromatography (SiO_2; eluent: 20% EtOAc in petroleum-ether), to give the compound **4**.

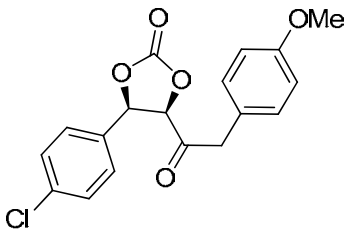
cis-4-(4-Chlorophenyl)-5-(2-phenylacetyl)-1,3-dioxolan-2-one ***4a***



Acording to the general procedure for the isomerization of carbonates, starting from 4-benzylidene-5-((4-chlorophenyl)-hydroxymethyl)-1,3-dioxolan-2-one **3a** (24.0 mg, 0.08 mmol) and DIPEA (2.2 mg, 3.0 µL, 0.04 mmol), after purification by dry-flash chromatography (SiO_2; eluent: 20% EtOAc in petroleum-ether), 21.6 mg (80%) of the title compound **4a** was obtained as white crystals.

Physical data for **4a** 142-143 ^{o}C; FT-IR (KBr, cm^{-1}): 3064, 2934, 1808, 1733, 1600, 1494, 1335, 1174, 1154, 1057. ^{1}H NMR ($CDCl_3$): 7.38 (d, *J* = 8.4 Hz, 2H), 7.26 - 7.22 (m, 3H), 7.16 (d, *J* = 8.4 Hz, 2H), 6.74 – 6.69 (m, 2H), 5.90 (d, *J* = 8.7 Hz, 1H), 5.34 (d, *J* = 8.7 Hz, 1H), 3.54 (d, *J* = 16.9 Hz, 1H), 3.29 (d, *J* = 16.9 Hz, 1H). ^{13}C NMR ($CDCl_3$): 201.0 (C), 153.3 (C), 136.2 (C), 130.5 (C), 130.4 (C), 129.5 (CH), 129.4 (CH), 128.7 (CH), 127.7 (CH), 127.6 (CH), 81.7 (CH), 78.8 (CH), 47.2 (CH_2). HRMS (ESI): calcd. for [$C_{17}H_{13}ClO_4$ + NH_4^+]: 334.0502, found for [M+NH_4]$^+$: 334.0831.

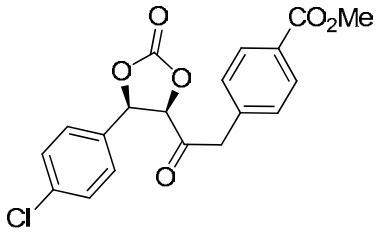
cis-4-(4-Chlorophenyl)-5-(2-(4-methoxyphenyl)acetyl)-1,3-dioxolan-2-one ***4b***



Acording to the general procedure for the isomerization of carbonates, starting from 4-((4-chlorophenyl)(hydroxy)methyl)-5-(4-methoxybenzylidene)-1,3-dioxolan-2-one **3c** (13.8 mg, 0.04 mmol) and DIPEA (2.2 mg, 3.0 µL, 0.04 mmol), after purification by dry-flash chromatography (SiO_2; eluent: 30% EtOAc in petroleum-ether), 10.8 mg (78%) of the title compound **4b** was obtained as white crystals.

Physical data for **4b** 121-123 ^{o}C; FT-IR (KBr, cm^{-1}): 2930, 2832, 1819, 1728, 1515, 1331, 1249, 1173, 1063, 841, 755. ^{1}H NMR ($CDCl_3$):7.36 (d, *J* = 8.3 Hz, 2H), 7.15 (d, *J* = 8.3 Hz, 2H), 6.77 (d, *J* = 8.5 Hz, 2H), 6.64 (d, *J* = 8.5 Hz, 2H), 5.88 (d, *J* = 8.8 Hz, 1H), 5.32 (d, *J* = 8.8 Hz, 1H), 3.77 (s, 3H), 3.47 (d, *J* = 17.0 Hz, 1H), 3.25 (d, *J* = 17.0 Hz, 1H). ^{13}C NMR ($CDCl_3$): 201.2 (C), 159.0 (C), 153.3 (C), 136.1 (C), 130.5 (CH), 129.3 (CH), 127.7 (CH), 127.5 (C), 122.3 (C), 114.2 (CH), 81.7 (CH), 78.8 (CH), 55.2 (CH_3), 46.4 (CH_2). HRMS (ESI): calcd. for [$C_{18}H_{15}ClO_5$ + NH_4^+]: 364.0946, found for [M+NH_4]$^+$: 364.0933.

cis-Methyl 4-((4-chlorophenyl)-2-oxo-1,3-dioxolan-4-yl)-2-oxoethyl)benzoate ***4c***



Acording to the general procedure for the isomerization of carbonates, starting from methyl 4-((4-chlorophenyl)(hydroxy)methyl)-2-oxo-1,3-dioxolan-4-ylidene)methyl) benzoate **3e** (6.0 mg, 0.016 mmol) and DIPEA (1.0 mg, 1.4 µL, 0.008 mmol), after purification by dry-flash chromatography (SiO_2; eluent: 30% EtOAc in petroleum-ether), 4.1 mg (68%) of the title compound **4c** was obtained as viscous oil.

Physical data for **4c:** FT-IR (KBr, cm^{-1}): 3094, 2953, 1810, 1717, 1612, 1282, 1165, 1060, 766. ^{1}H NMR ($CDCl_3$): 7.90 (d, *J* = 8.4 Hz, 2H), 7.39 (d, *J* = 8.4 Hz, 2H), 7.18 (d, *J* = 8.8 Hz, 2H), 6.74 (d, *J* = 8.6 Hz, 2H), 5.94 (d, *J* = 8.8 Hz, 1H), 5.35 (d, *J* = 8.8 Hz, 1H), 3.90 (s, 3H), 3.48 (d, *J* = 17.1 Hz, 1H), 3.37 (d, *J* = 17.1 Hz, 1H). ^{13}C NMR ($CDCl_3$): 200.6 (C), 166.7 (C), 153.2 (C), 136.4 (C), 135.7 (C), 130.4 (C), 129.9 (CH),

129.5 (CH), 129.4 (CH), 128.5 (C), 127.7 (CH), 81.9 (CH), 78.6 (CH), 52.1 (CH_2), 47.0 (CH_3). HRMS (ESI): calcd. for [$C_{19}H_{15}ClO_6$ + NH_4^+]: 392.0557, found for [M+NH_4]$^+$: 392.0892.

References

[i] For description of the technique of dry-flash chromatography, see: a) Harwood, L. M. *Aldrichimica Acta* **1985**, *18*, 25; b) *Vogel's Textbook of Practical Organic Chemistry*, Longman Scientific&Technical, 5th edition, London, 1989, p. 220; c) A recent account which includes some improvements of the separation technique: Pedersen, D. S.; Rosenbohm, C. *Synthesis* **2001**, 2431.

[ii] Perrin, D. D.; Armarego, W. L. F. *Purification of Laboratory Chemicals*, 3rd edition, Pergamon Press, **1988**.

[iii] Bigovic, M., Maslak, V., Tokic-Vujosevic, Z., Saicic, R. N. *Org. Lett.* **2011**, *13*, 4720.

VM-MB-7-24A
Solvent: cdcl3
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1st pulse arrayed
2nd pulse 90.0 degrees
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Width 4600.0 Hz
Arrayed repetitions
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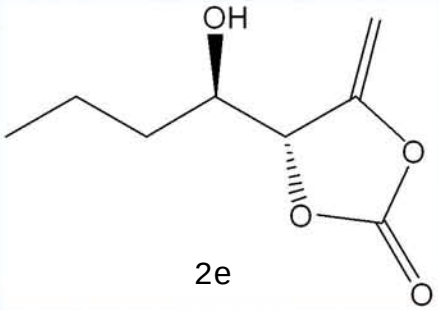
DATA PROCESSING
FT size 16384
Total time 1 minute



2e

VM-MB-7-24 A

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Ambient temperature
GEMINI-200 "nmr"

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1st pulse arrayed
2nd pulse 81.8 degrees
Acq. time 1.067 sec
Width 15000.0 Hz
Arrayed repetitions
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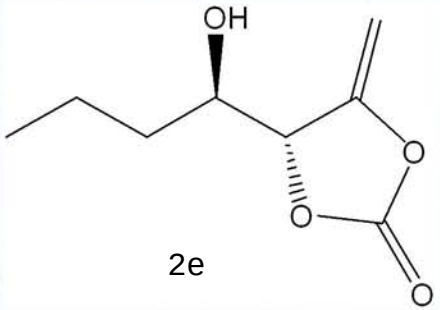
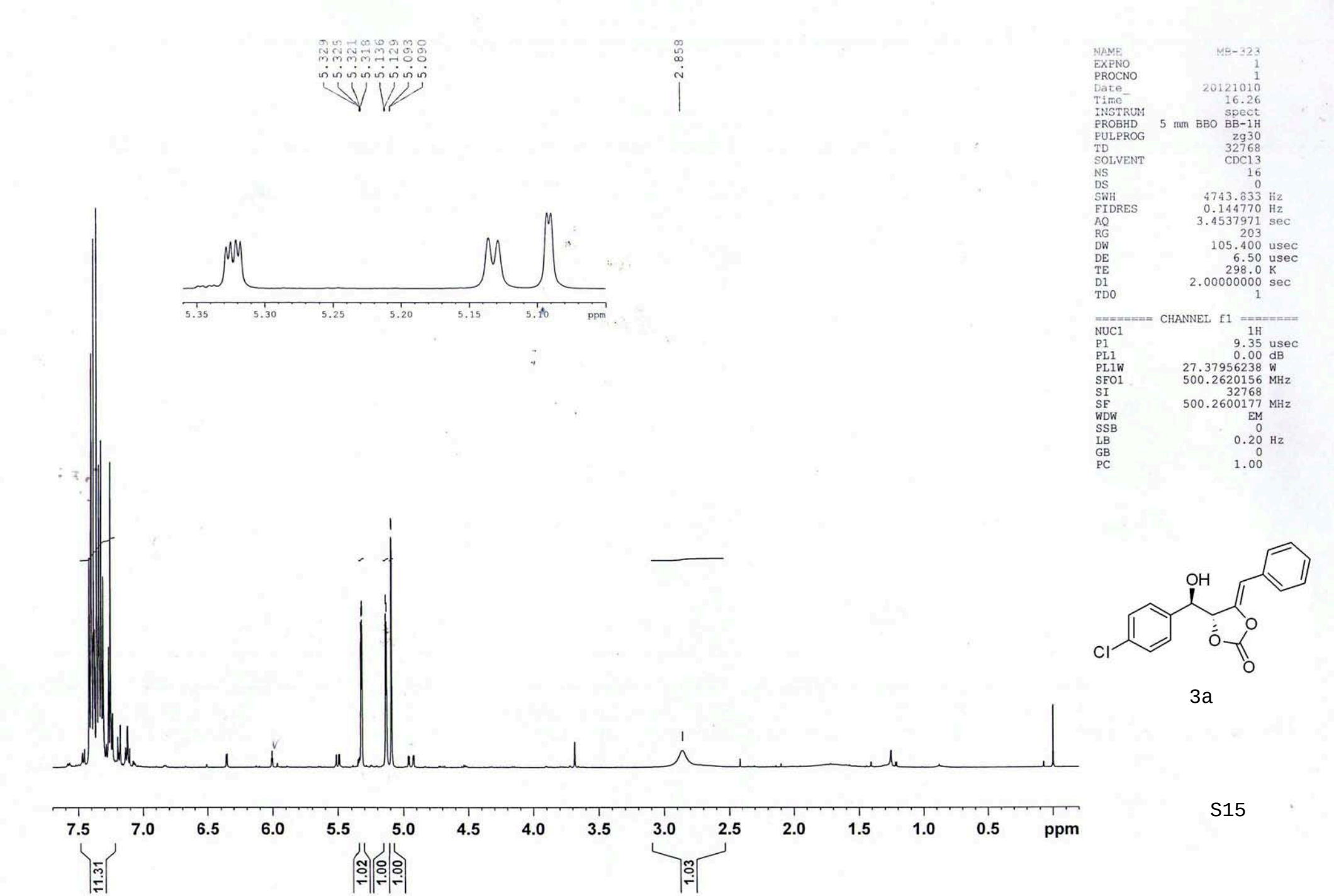
DECOUPLE H1, 199.9712807 MHz
Power 0 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 32768
Total time 36 minutes



2e



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RG	203
DW	105.400 usec
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TE	298.0 K
D1	2.00000000 sec
TD0	1
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PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2620156 MHz
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SF	500.2600177 MHz
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GB	0
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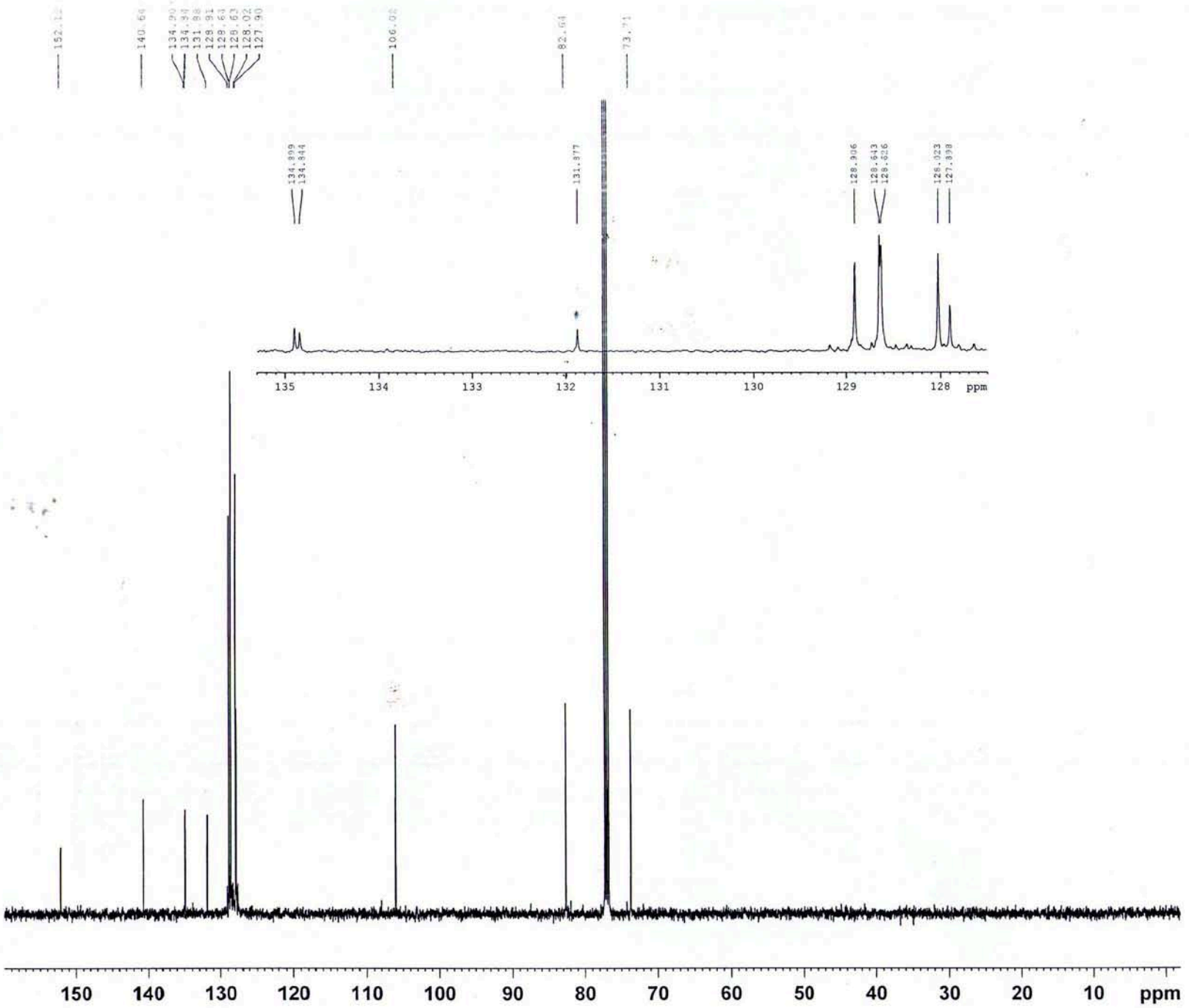
3a



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PROCNO	1
Date_	20121010
Time	16.32
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TD	32768
SOLVENT	CDCl3
NS	332
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	0.5505524 sec
RG	1030
DW	16.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.50 usec
PL1	3.00 dB
PL1W	32.22848892 W
SFO1	125.8043140 MHz
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NUC2	1H
PCPD2	80.00 usec
PL2	1.20 dB
PL12	18.40 dB
PL13	18.40 dB
PL2W	20.76952171 W
PL12W	0.39575511 W
PL13W	0.39575511 W
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WDW	EM
SSB	0
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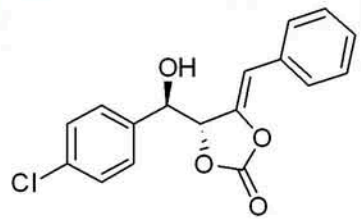
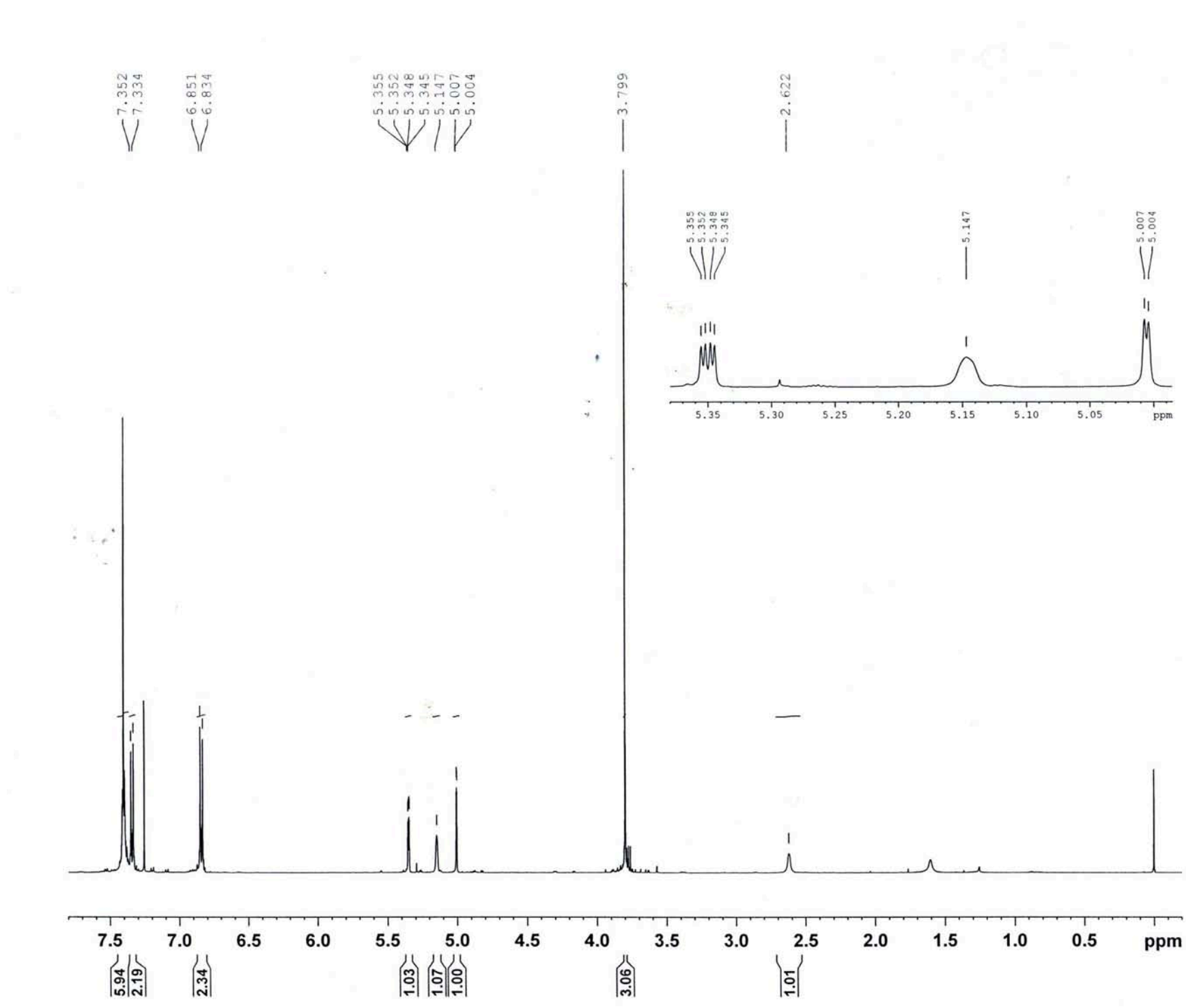


3a



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SOLVENT	CDCl3
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DS	0
SWH	4770.992 Hz
FIDRES	0.145599 Hz
AQ	3.4341364 sec
RG	256
DW	104.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
TD0	1
========	CHANNEL f1 ========
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P1	9.35 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2621162 MHz
SI	32768
SF	500.2600163 MHz
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GB	0
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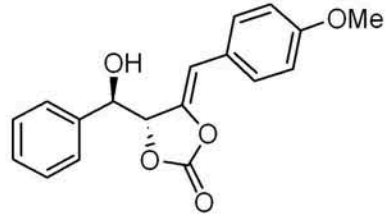
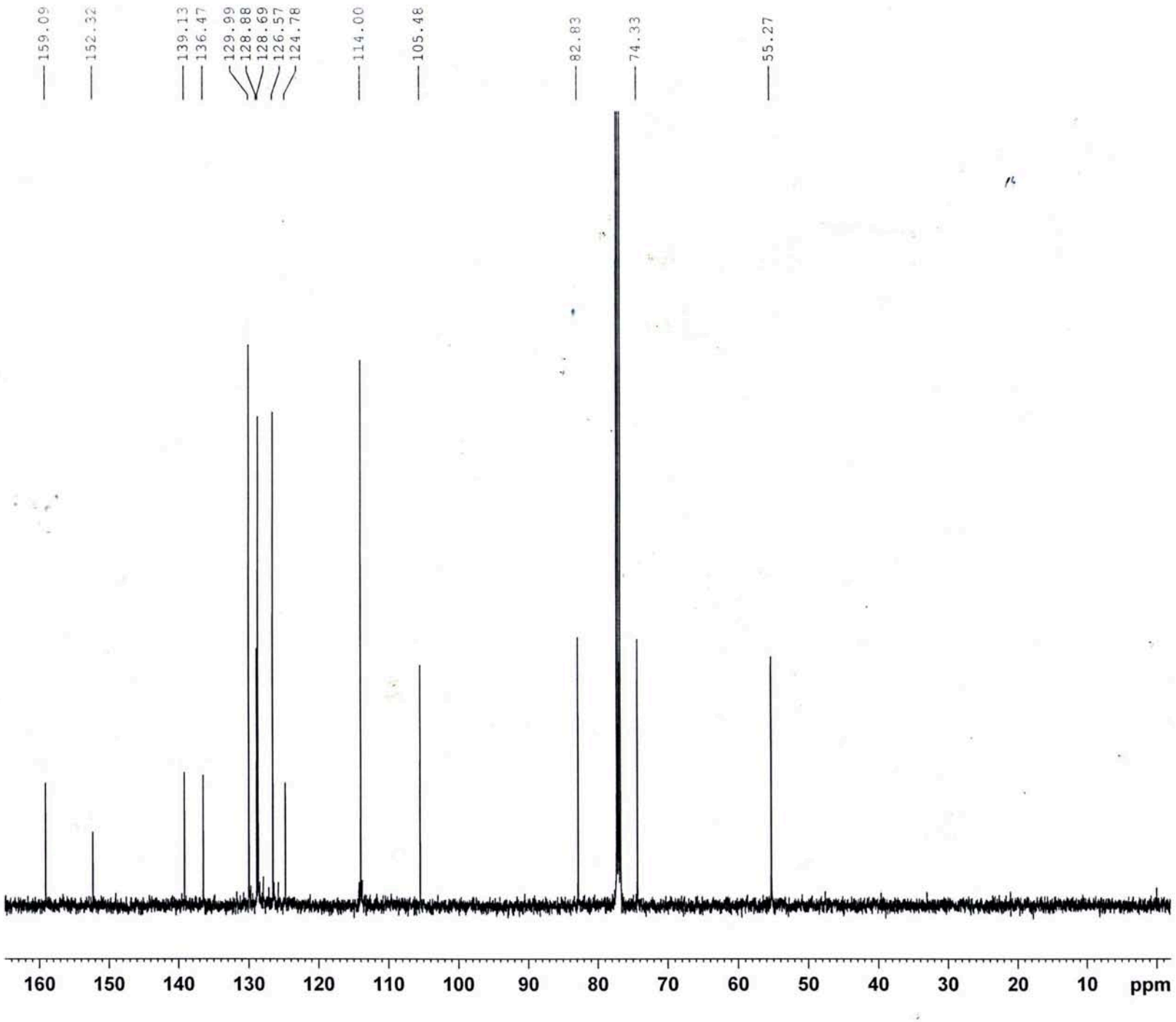


3b



NAME	MB-266
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PROCNO	1
Date_	20120629
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PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
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FIDRES	0.908261 Hz
AQ	0.5505524 sec
RG	1030
DW	16.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.50 usec
PL1	3.00 dB
PL1W	32.22848892 W
SFO1	125.8043140 MHz
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NUC2	1H
PCPD2	80.00 usec
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PL12W	0.39575511 W
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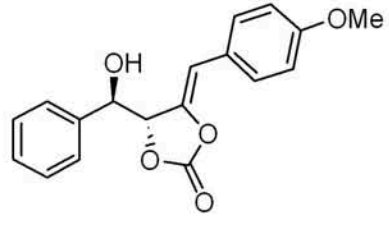

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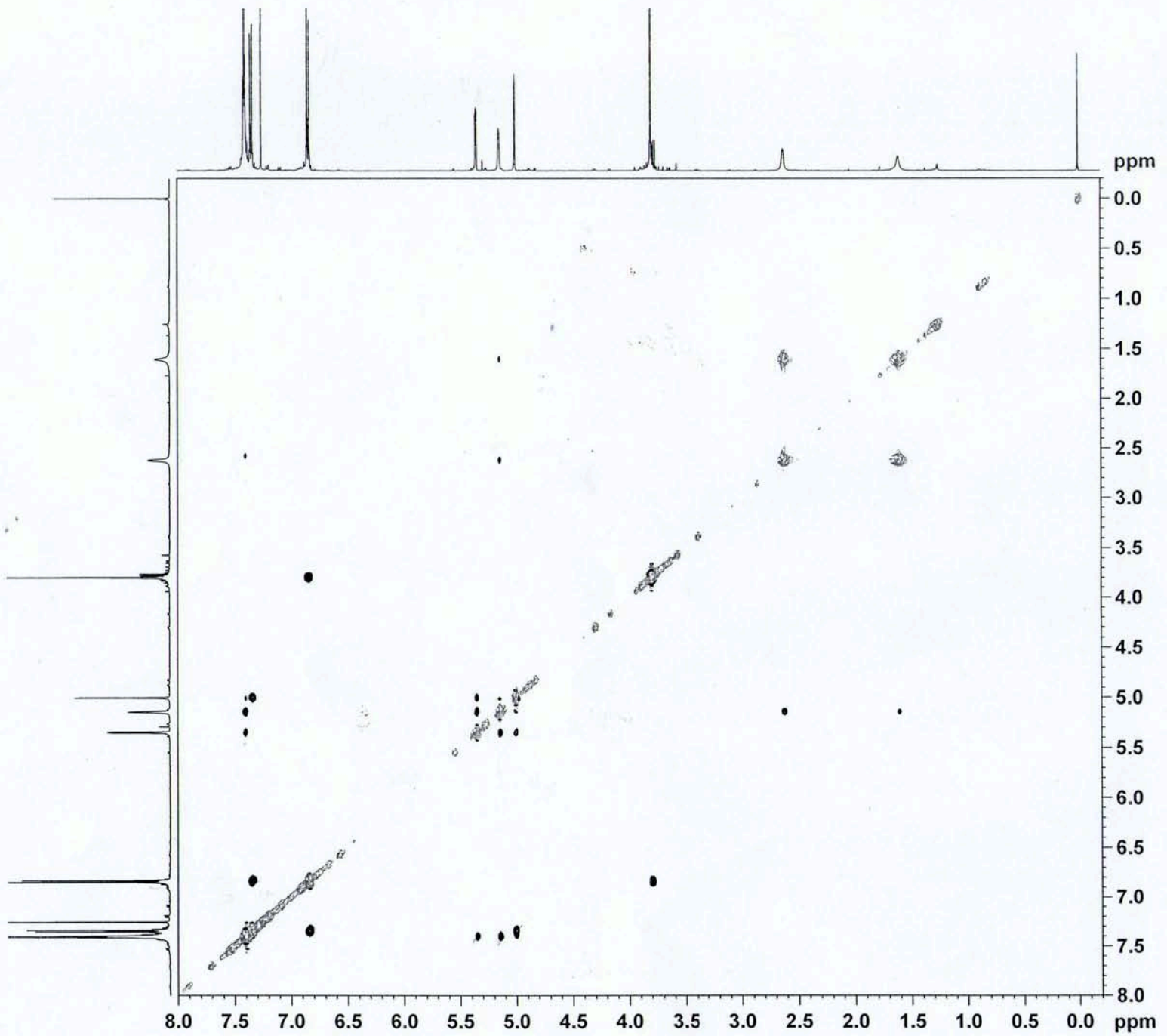

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LB	0.00 Hz
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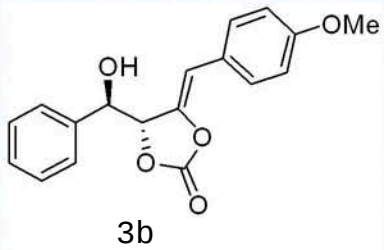
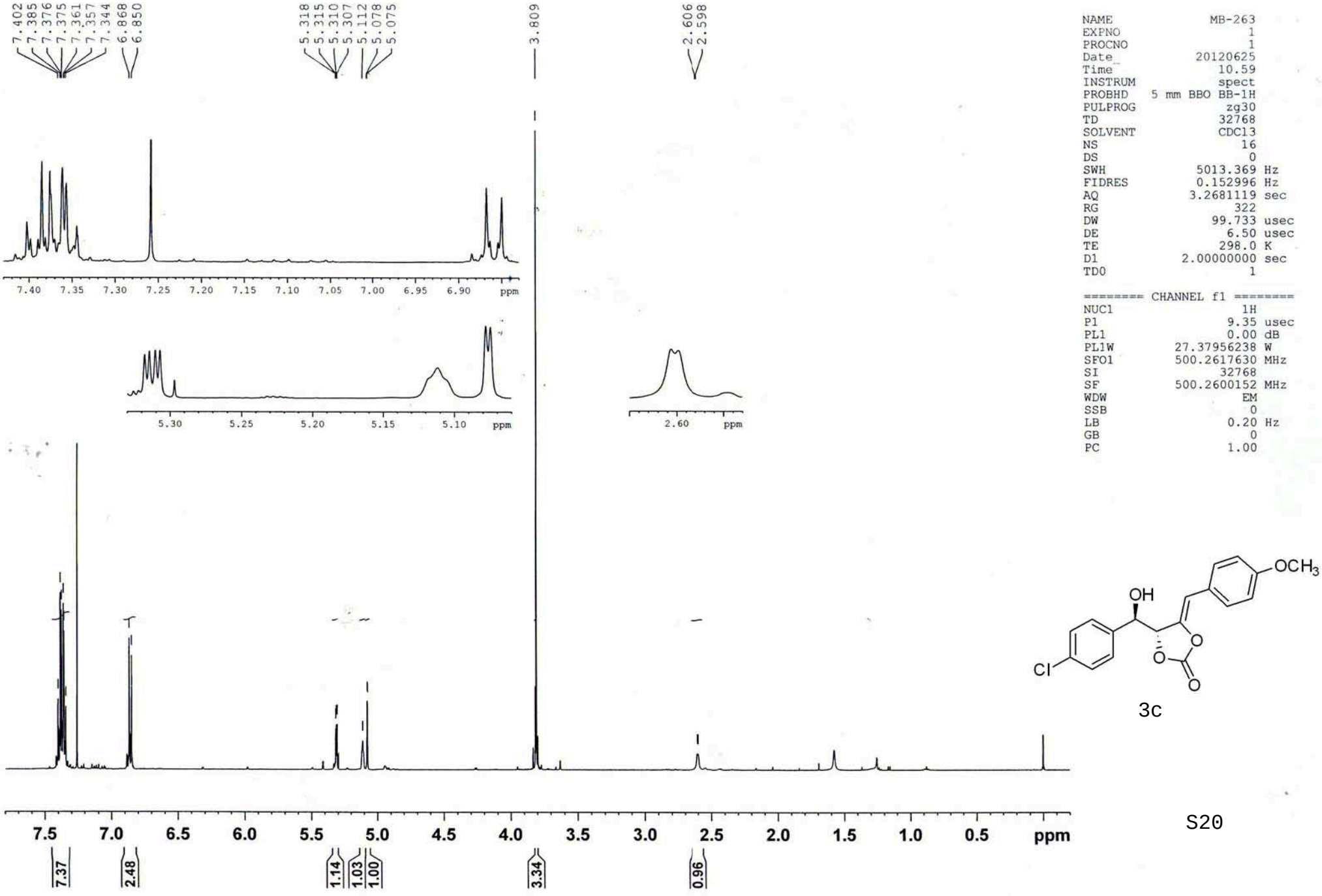


3b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LB 0.20 Hz
GB 0
PC 1.00
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OH
Cl
O
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5.30 5.25 5.20 5.15 5.10 ppm
2.60 ppm
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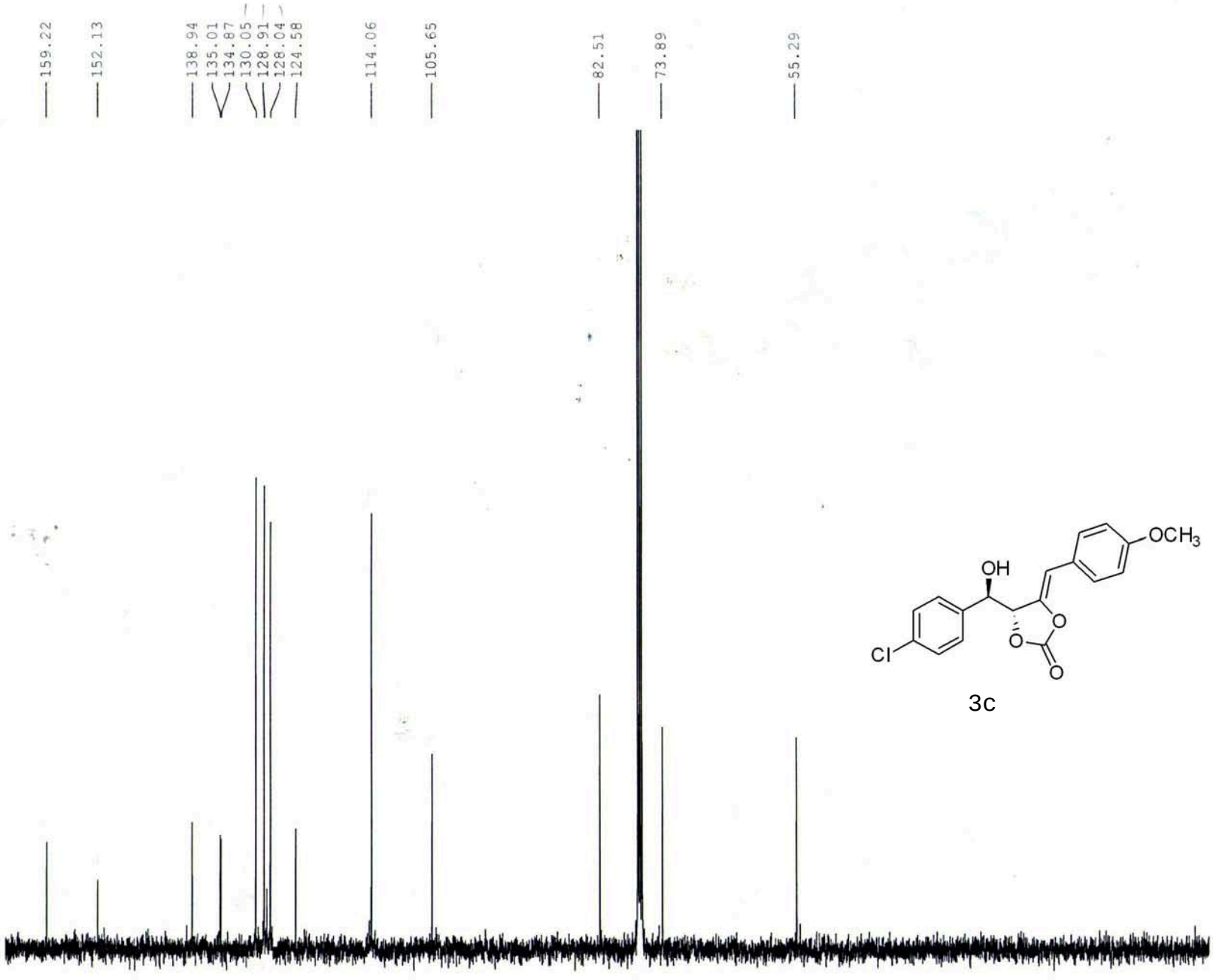
1.03
1.00
3.34
0.96

3c



3c

NAME	MB-263
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PL1	3.00 dB
PL1W	32.22848892 W
SFO1	125.8043140 MHz
========	CHANNEL f2 ========
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NUC2	1H
PCPD2	80.00 usec
PL2	1.20 dB
PL12	18.40 dB
PL13	18.40 dB
PL2W	20.76952171 W
PL12W	0.39575511 W
PL13W	0.39575511 W
SFO2	500.2617629 MHz
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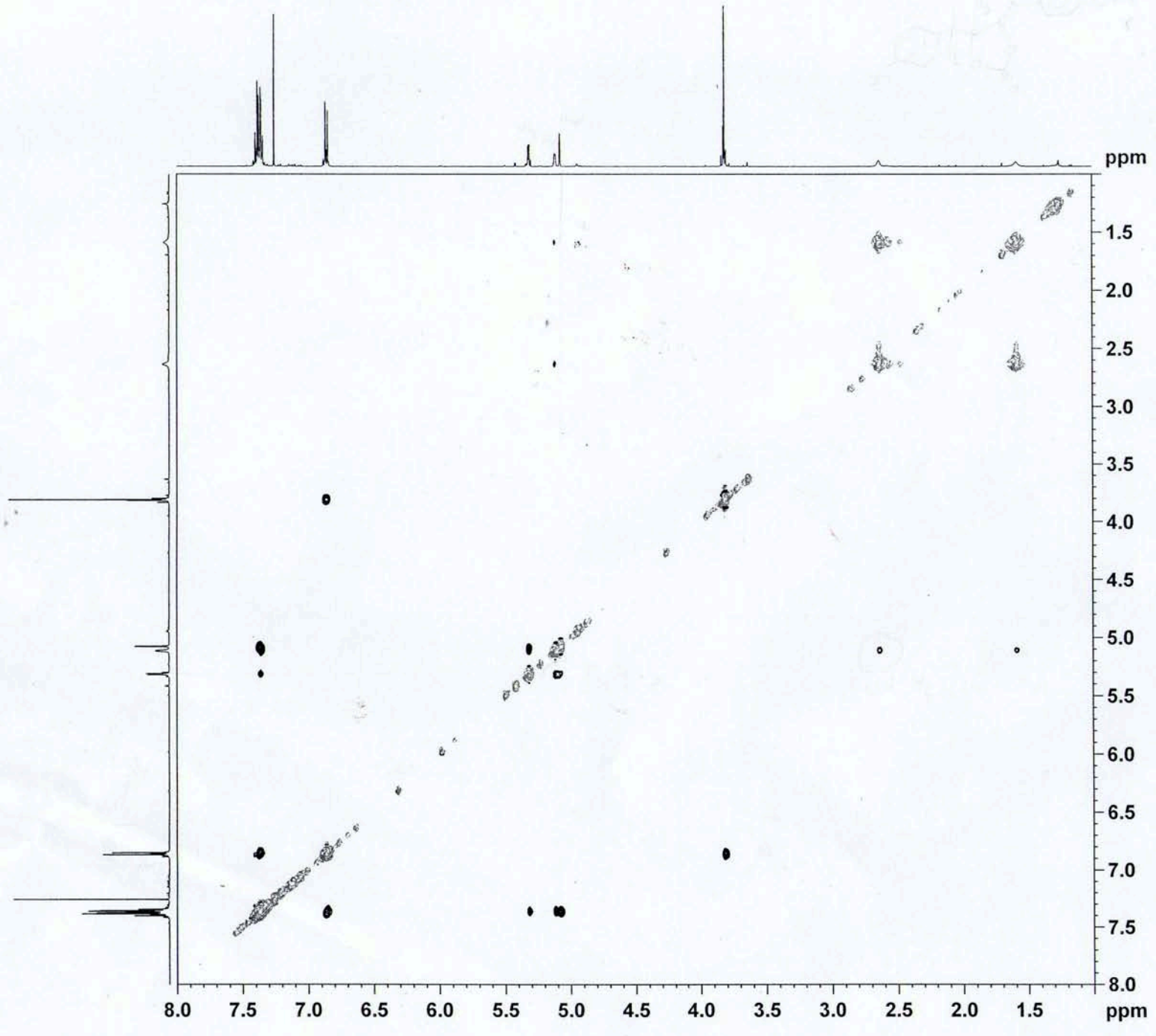
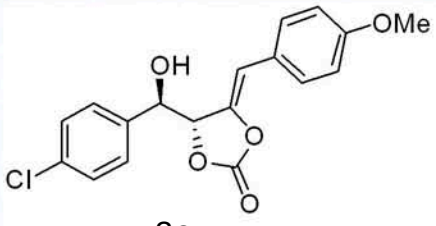
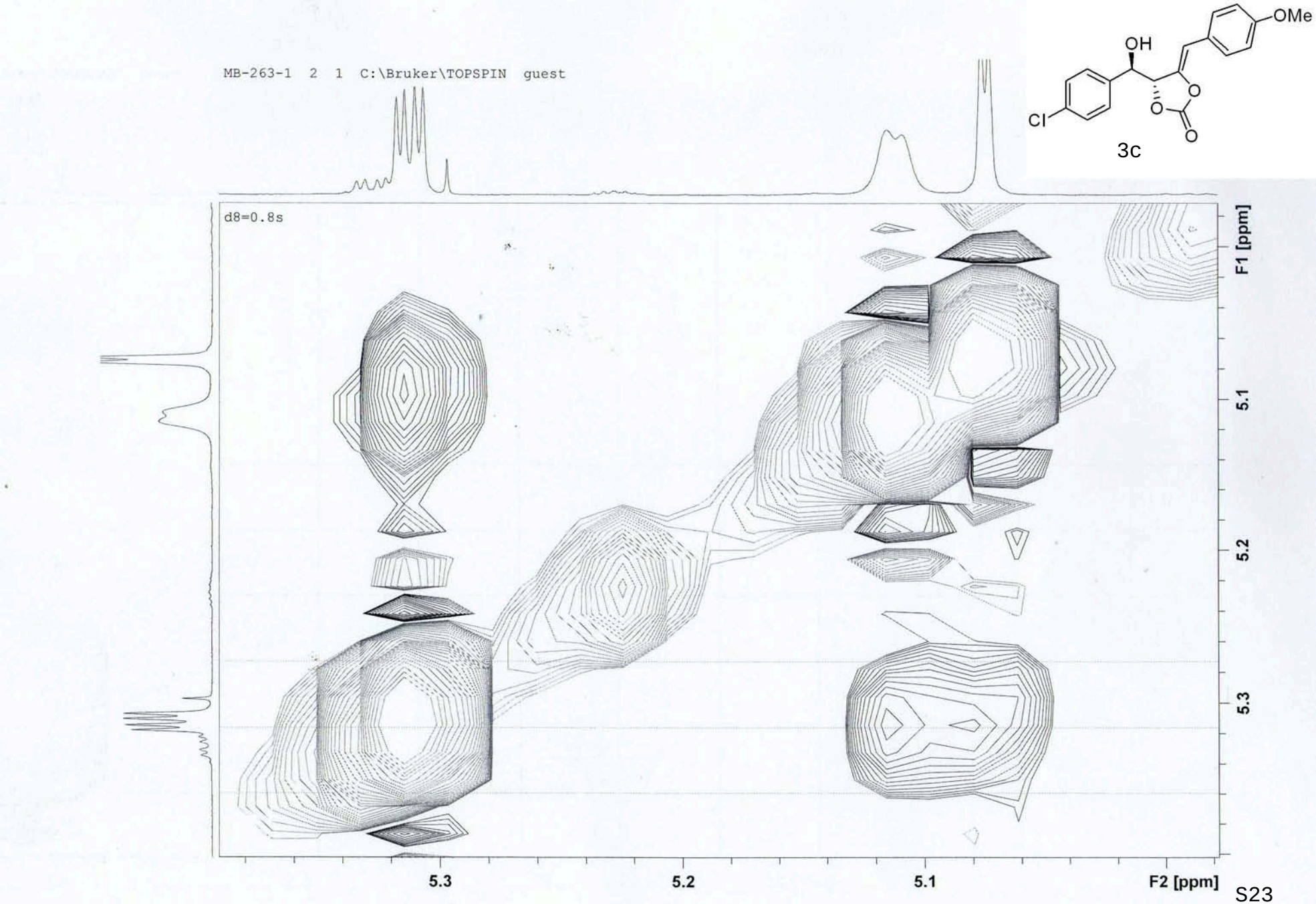
SF	125.7904805 MHz
WDW	EM
SSB	0
LB	1.50 Hz
GB	0
PC	1.40

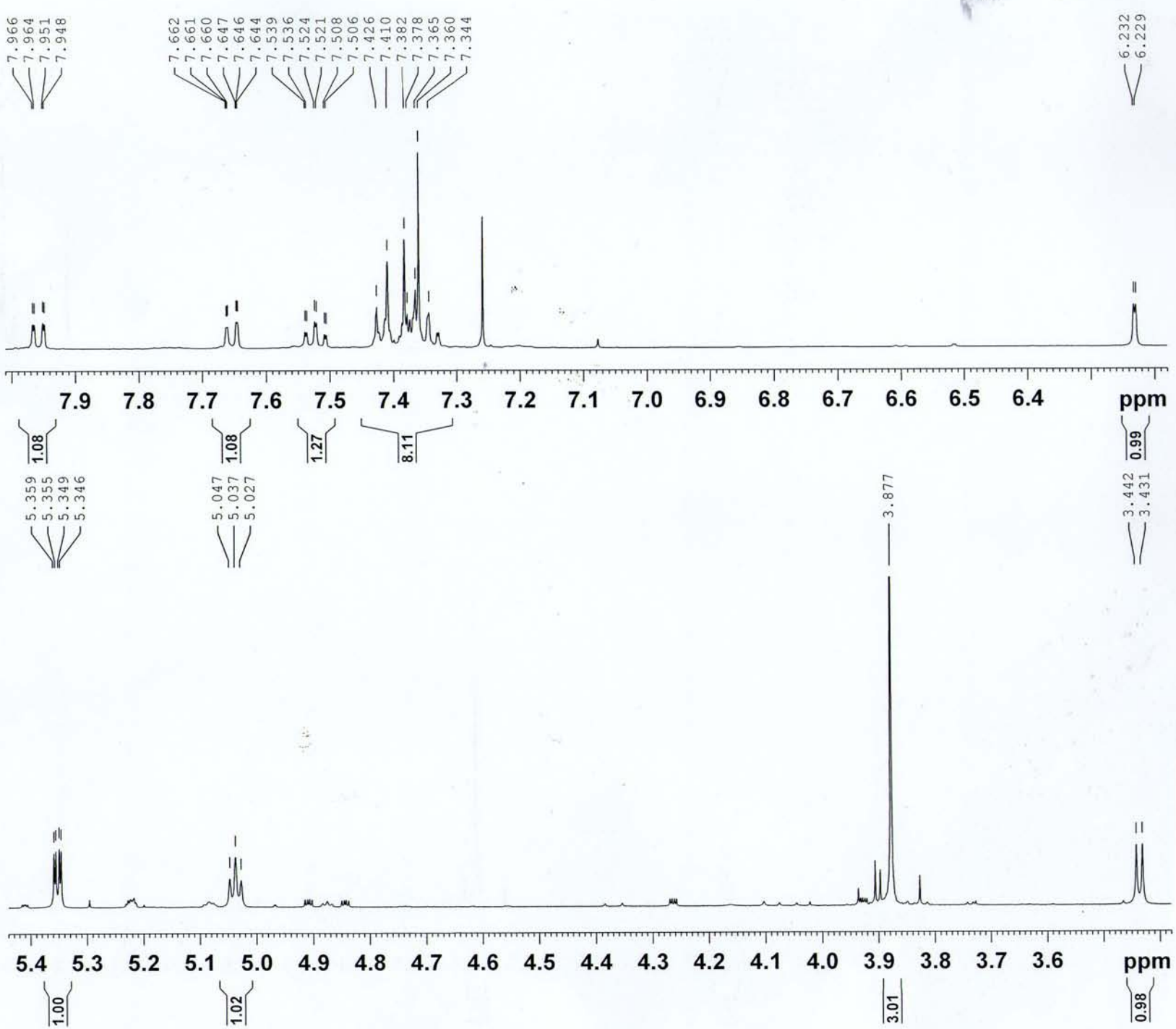


NAME	MB-263-1
EXPNO	2
PROCNO	1
Date_	20120628
Time	11.13
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	noesygpph
TD	1024
SOLVENT	CDCl3
NS	8
DS	16
SWH	4629.629 Hz
FIDRES	4.521122 Hz
AQ	0.1106420 sec
RG	322
DW	108.000 usec
DE	6.50 usec
TE	298.0 K
D0	0.00009610 sec
D1	2.00000000 sec
D8	0.80000001 sec
D16	0.00020000 sec
IN0	0.00021600 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	9.35 usec
P2	18.70 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2619510 MHz
====== GRADIENT CHANNEL ======	
GPNAM1	SINE.100
GPZ1	40.00 %
P16	1000.00 usec
ND0	1
TD	164
SFO1	500.262 MHz
FIDRES	28.229416 Hz
SW	9.254 ppm
FnMODE	States-TPPI
SI	512
SF	500.2600112 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	512
MC2	States-TPPI
SF	500.2600115 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

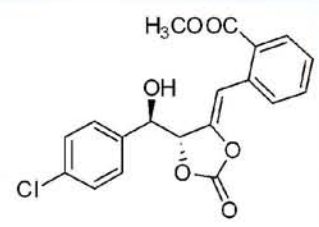


3c

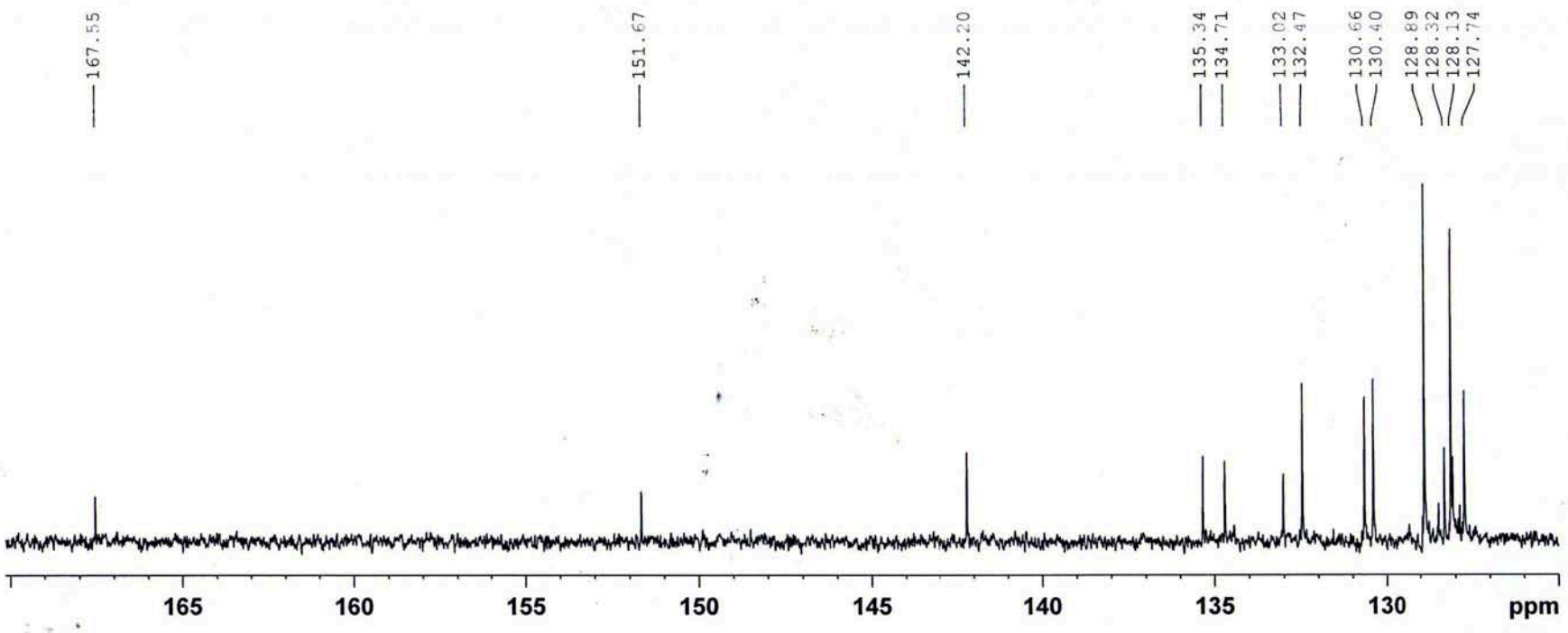

MB-263-1 2 1 C:\Bruker\TOPSPIN guest
d8=0.8s
OH
OMe
Cl
O
O
O
3c
F1 [ppm]
5.1
5.2
5.3
5.3
5.2
5.1
F2 [ppm]

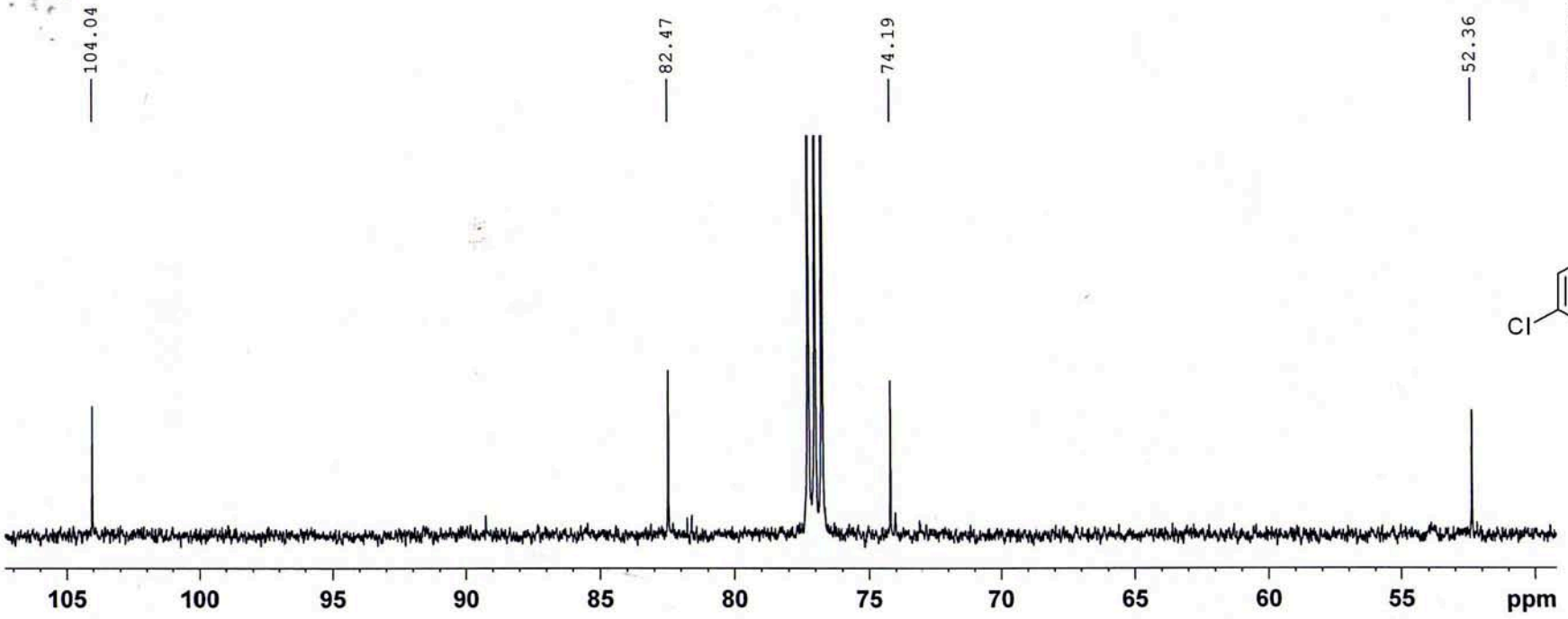



```
Current Data Parameters
NAME                  MB-315-2
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameter
Date_                 20121010
Time                     11.52
INSTRUM                  spect
PROBHD    5 mm BBO BB-1H
PULPROG                   zg30
TD                       32768
SOLVENT                  CDCl3
NS                          16
DS                           0
SWH                 4870.130 H
FIDRES              0.148625 H
AQ                  3.3642313 s
RG                         181
DW                   102.667 u
DE                      6.50 u
TE                     298.0 K
D1                 2.00000000 s
TD0                          1

======== CHANNEL f1 ======
NUC1                        1H
P1                       9.35 u
PL1                      0.00 d
PL1W               27.37956238 W
SFO1               500.2620828 M

F2 - Processing parameters
SI                       32768
SF                 500.2600154 M
WDW                         EM
SSB                          0
LB                        0.20 H
GB                           0
PC                        1.00
```

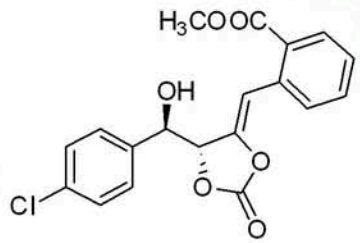


3d






```
NAME              MB-315-2
EXPNO                    2
PROCNO                   1
Date_             20121010
Time                 12.06
INSTRUM              spect
PROBHD   5 mm BBO BB-1H
PULPROG              zgpg30
TD                    32768
SOLVENT               CDCl3
NS                      517
DS                        4
SWH              29761.904 Hz
FIDRES           0.908261 Hz
AQ               0.5505524 sec
RG                     1030
DW                16.800 usec
DE                 6.50 usec
TE                 298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                   13C
P1                 11.50 usec
PL1                 3.00 dB
PL1W         32.22848892 W
SFO1        125.8043140 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                 1.20 dB
PL12               18.40 dB
PL13               18.40 dB
PL2W         20.76952171 W
PL12W         0.39575511 W
PL13W         0.39575511 W
SFO2        500.2620826 MHz
SI                  32768
SF          125.7904814 MHz
WDW                    EM
SSB                     0
LB                 1.50 Hz
GB                      0
PC                   1.40
```

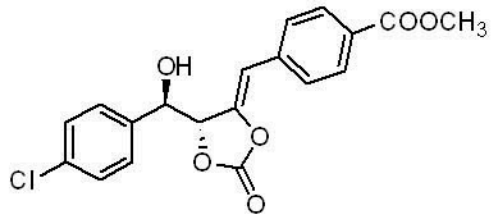


3d

MB-336

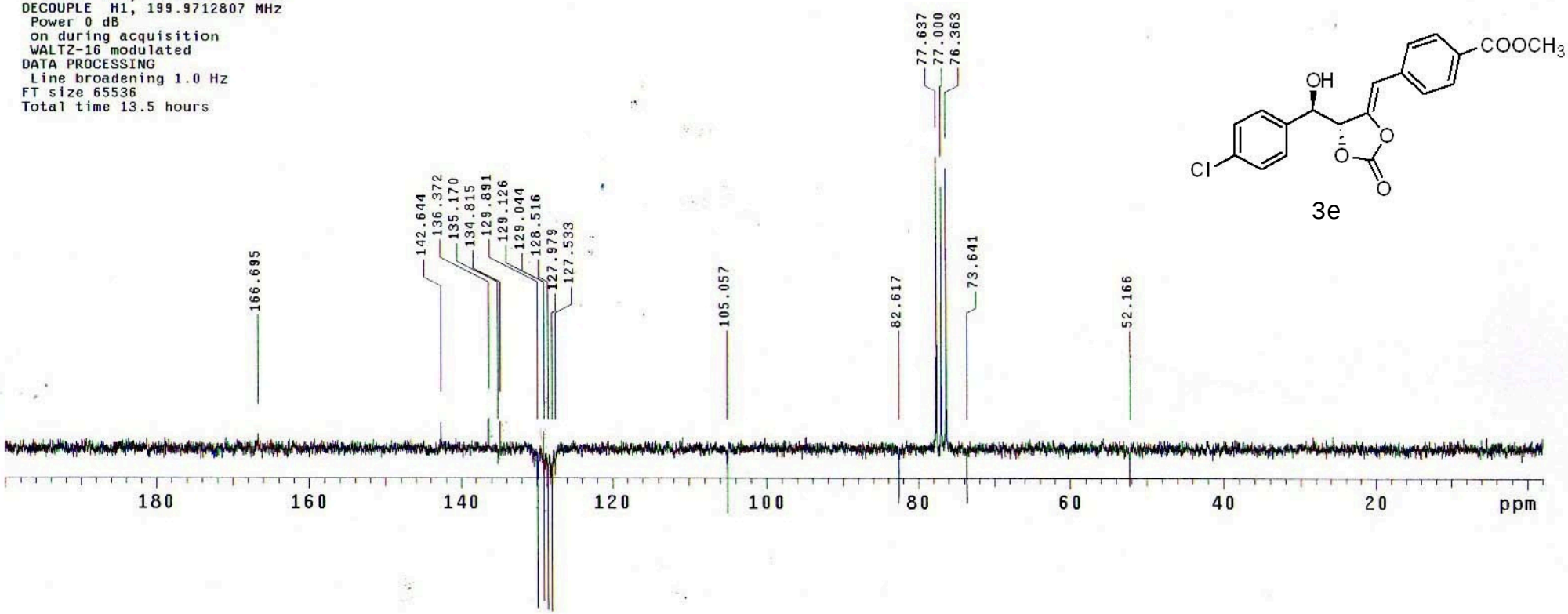
Solvent: cdcl3
Ambient temperature
File: hmb336
GEMINI-200 "nmr"

PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 90.0 degrees
Acq. time 1.440 sec
Width 4600.0 Hz
Arrayed repetitions
OBSERVE H1, 199.9710956 MHz
DATA PROCESSING
FT size 16384
Total time 5 minutes

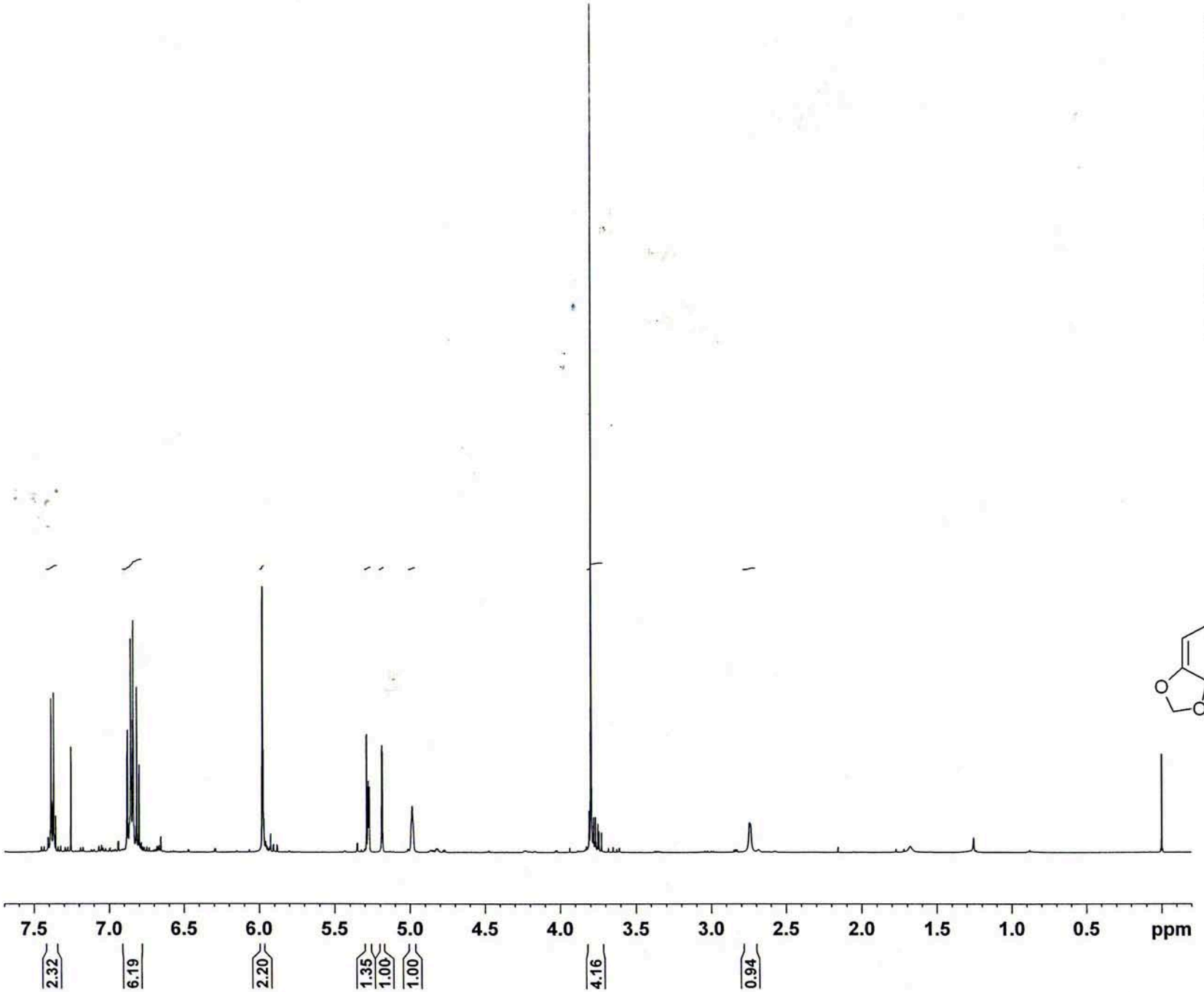


3e

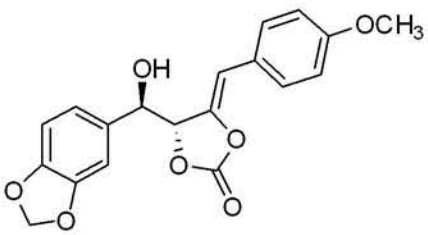
MB-336-2
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE: apt
Relax. delay arrayed
1st pulse arrayed
2nd pulse 122.7 degrees
Acq. time 2.000 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.2827780 MHz
DECOUPLE H1, 199.9712807 MHz
Power 0 dB
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 13.5 hours



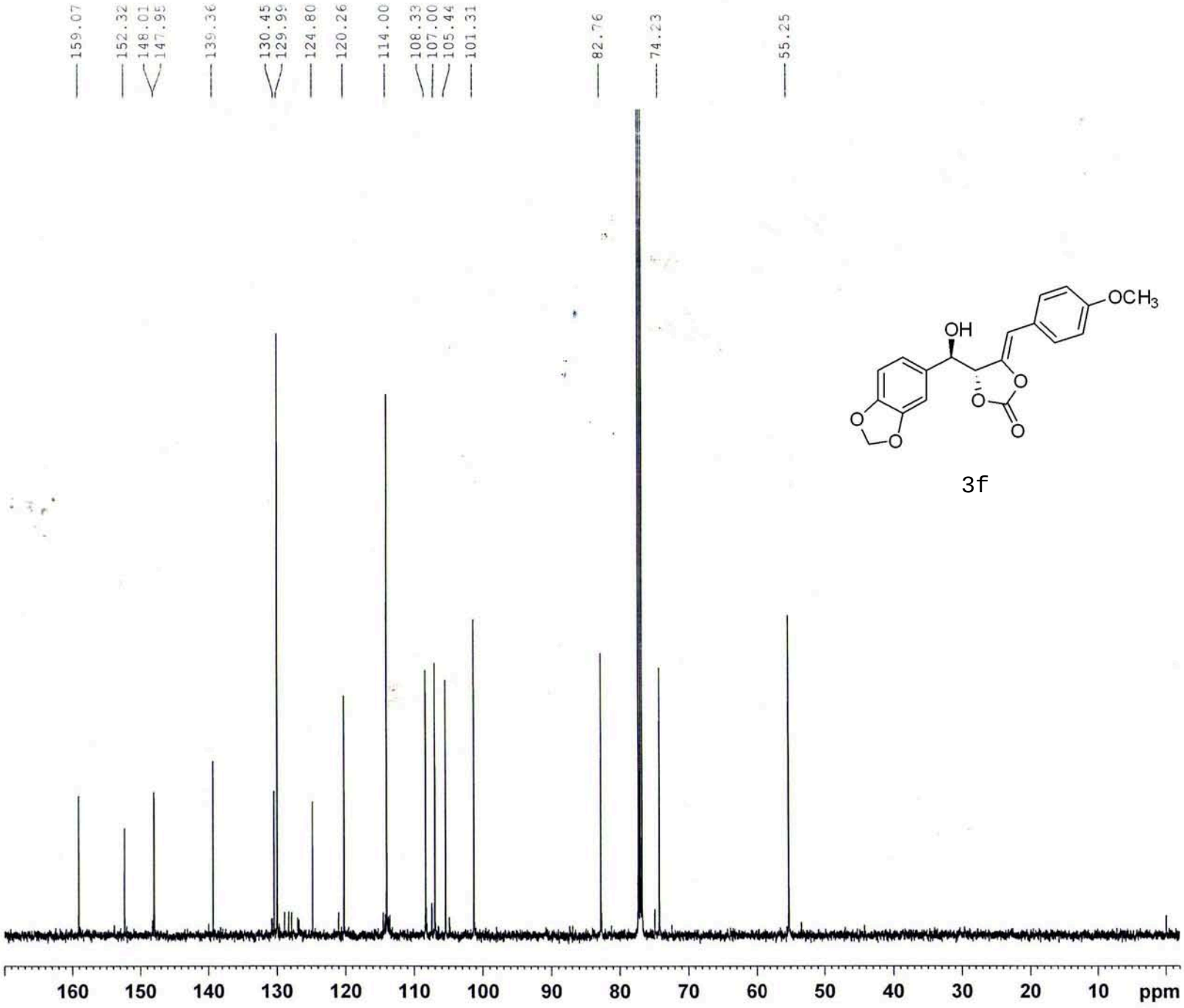
3e



NAME	MB-307-2
EXPNO	1
PROCNO	1
Date_	20120924
Time	11.00
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	4652.605 Hz
FIDRES	0.141986 Hz
AQ	3.5215178 sec
RG	101
DW	107.467 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	9.35 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2620531 MHz
SI	32768
SF	500.2600169 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.00

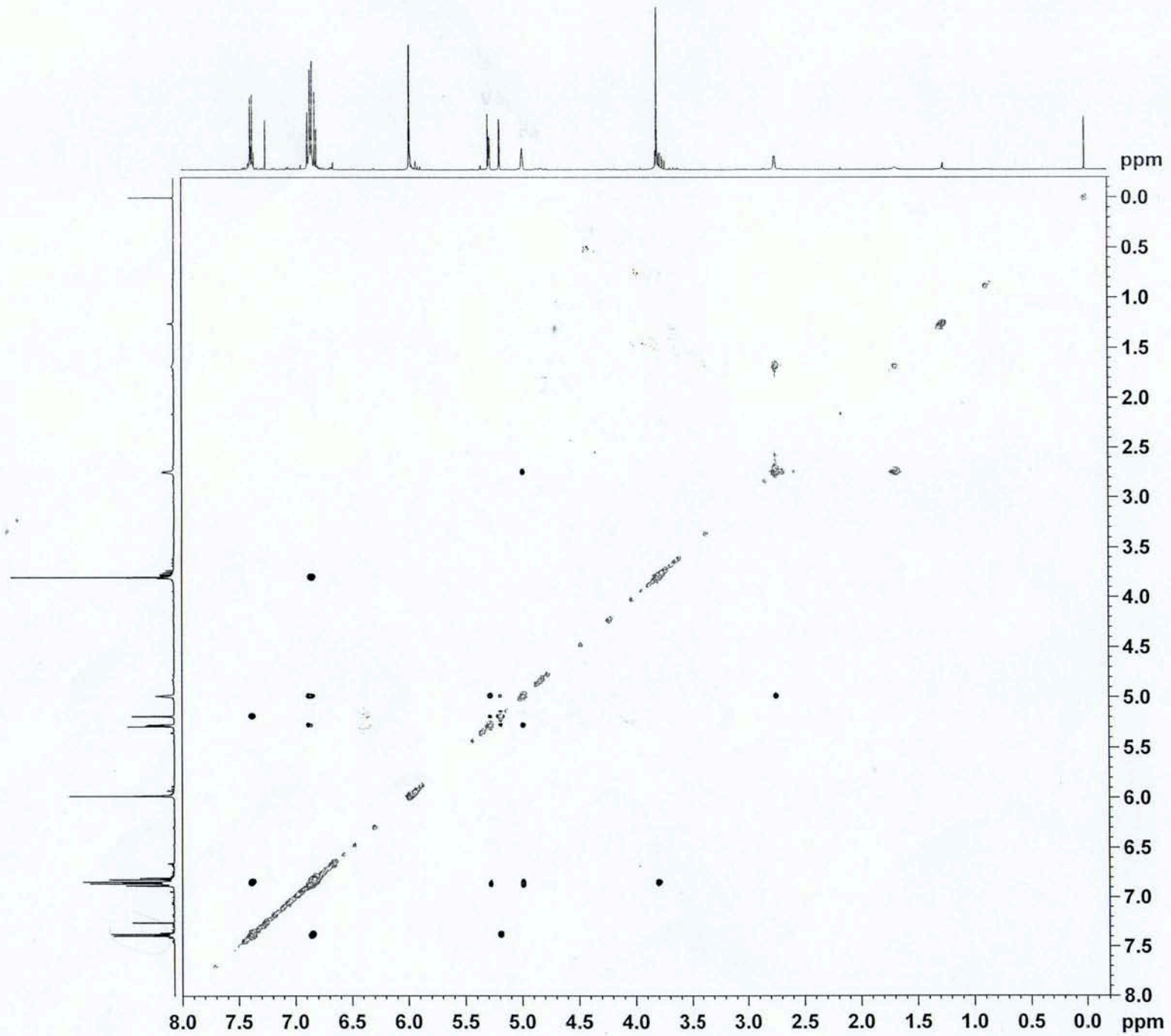


3f

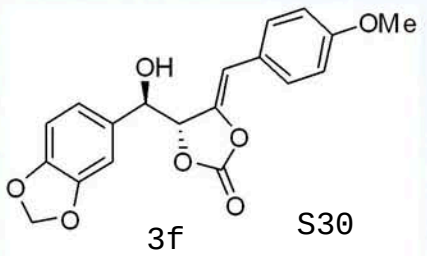


3f

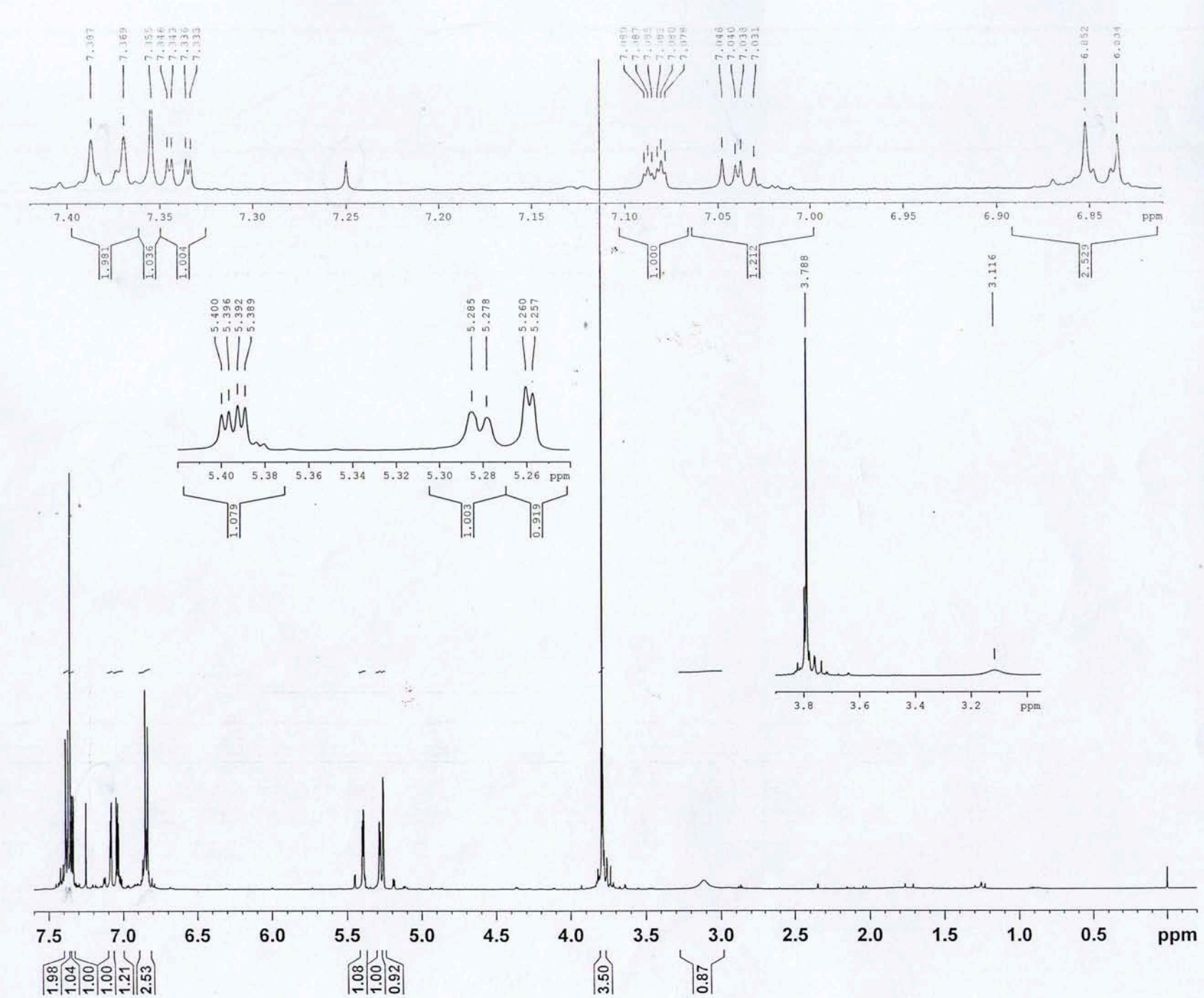
NAME	MB-307-2
EXPNO	2
PROCNO	1
Date_	20120924
Time	11.20
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	512
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	0.5505524 sec
RG	912
DW	16.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.50 usec
PL1	3.00 dB
PL1W	32.22848892 W
SFO1	125.8043140 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.20 dB
PL12	18.40 dB
PL13	18.40 dB
PL2W	20.76952171 W
PL12W	0.39575511 W
PL13W	0.39575511 W
SFO2	500.2620531 MHz
SI	32768
SF	125.7904847 MHz
WDW	EM
SSB	0
LB	1.50 Hz
GB	0
PC	1.40



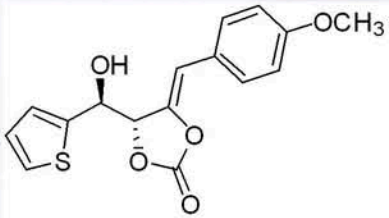
NAME	MB-307-2
EXPNO	4
PROCNO	1
Date_	20120924
Time	11.52
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	noesygpph
TD	1024
SOLVENT	CDCl3
NS	4
DS	16
SWH	4652.605 Hz
FIDRES	4.543560 Hz
AQ	0.1100959 sec
RG	101
DW	107.467 usec
DE	6.50 usec
TE	298.0 K
D0	0.00009556 sec
D1	2.00000000 sec
D8	1.00000000 sec
D16	0.00020000 sec
IN0	0.00021495 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	9.35 usec
P2	18.70 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2620531 MHz
====== GRADIENT CHANNEL =====	
GPNAM1	SINE.100
GPZ1	40.00 %
P16	1000.00 usec
ND0	1
TD	256
SFO1	500.2621 MHz
FIDRES	18.174170 Hz
SW	9.300 ppm
FnMODE	States-TPPI
SI	512
SF	500.2600130 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	512
MC2	States-TPPI
SF	500.2600131 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0



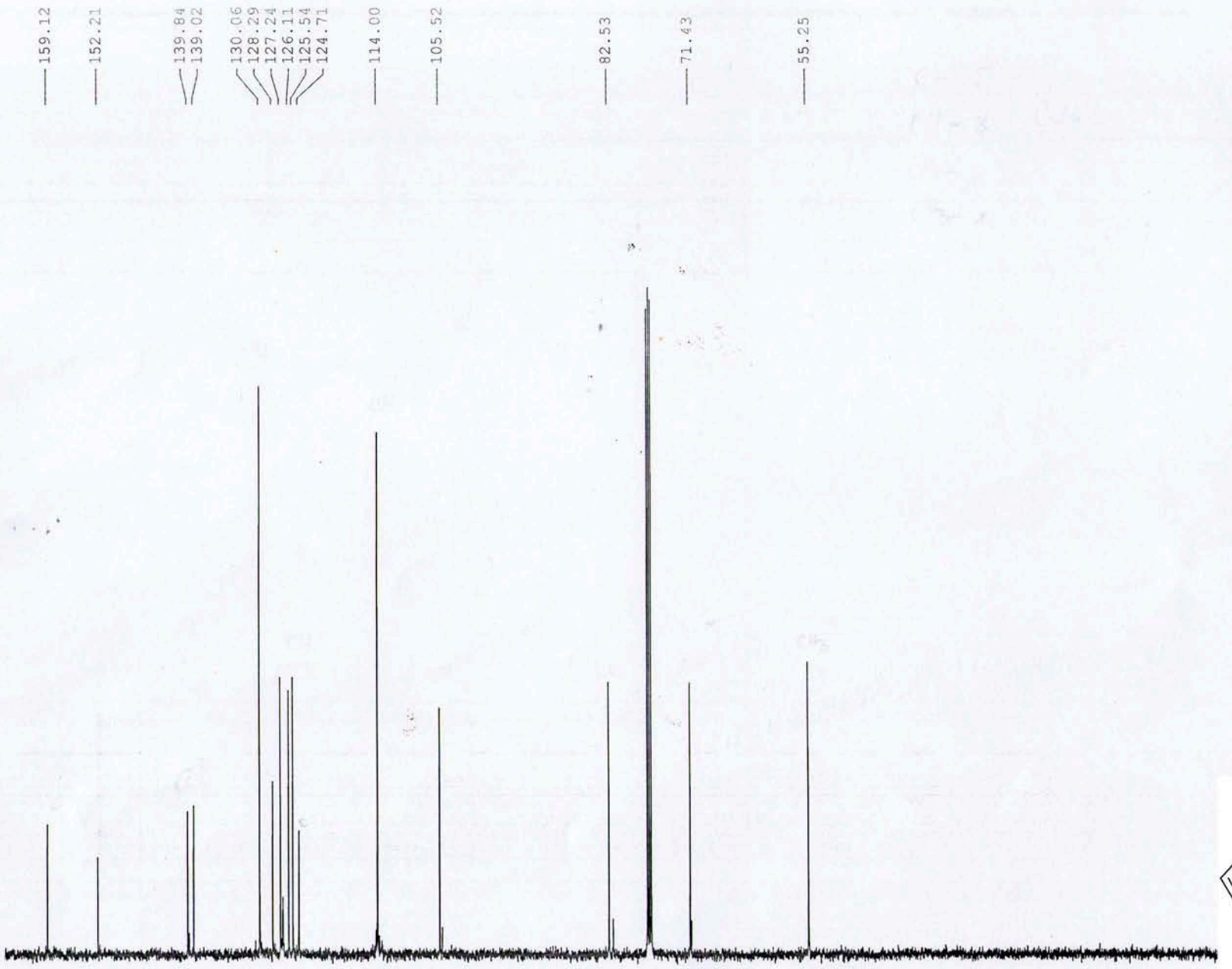
3f



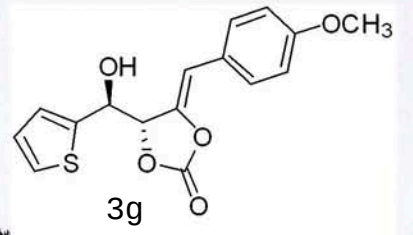
NAME	MB-322
EXPNO	1
PROCNO	1
Date_	20121010
Time	15.33
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	4746.835 Hz
FIDRES	0.144862 Hz
AQ	3.4516127 sec
RG	101
DW	105.333 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	9.35 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2621415 MHz
SI	32768
SF	500.2600196 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.00



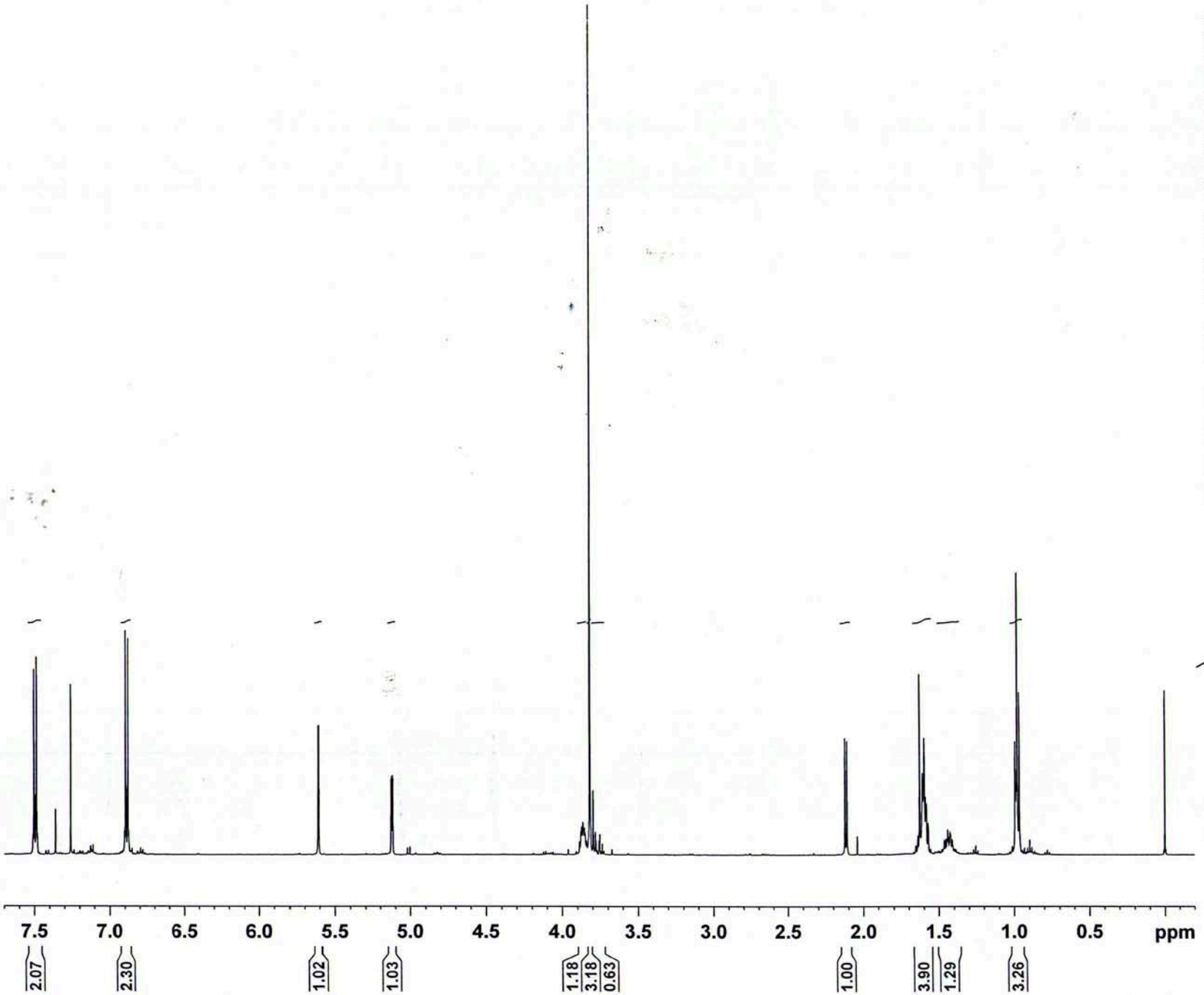
3g



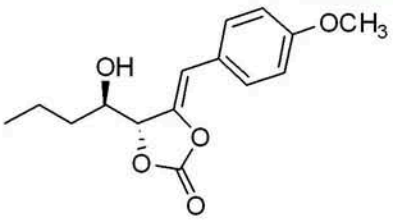
NAME	MB-322
EXPNO	2
PROCNO	1
Date_	20121010
Time	15.40
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	271
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	0.5505524 sec
RG	1030
DW	16.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.50 usec
PL1	3.00 dB
PL1W	32.22848892 W
SFO1	125.8043140 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.20 dB
PL12	18.40 dB
PL13	18.40 dB
PL2W	20.76952171 W
PL12W	0.39575511 W
PL13W	0.39575511 W
SFO2	500.2621416 MHz
SI	32768
SF	125.7904860 MHz
WDW	EM
SSB	0
LB	1.50 Hz
GB	0
PC	1.40



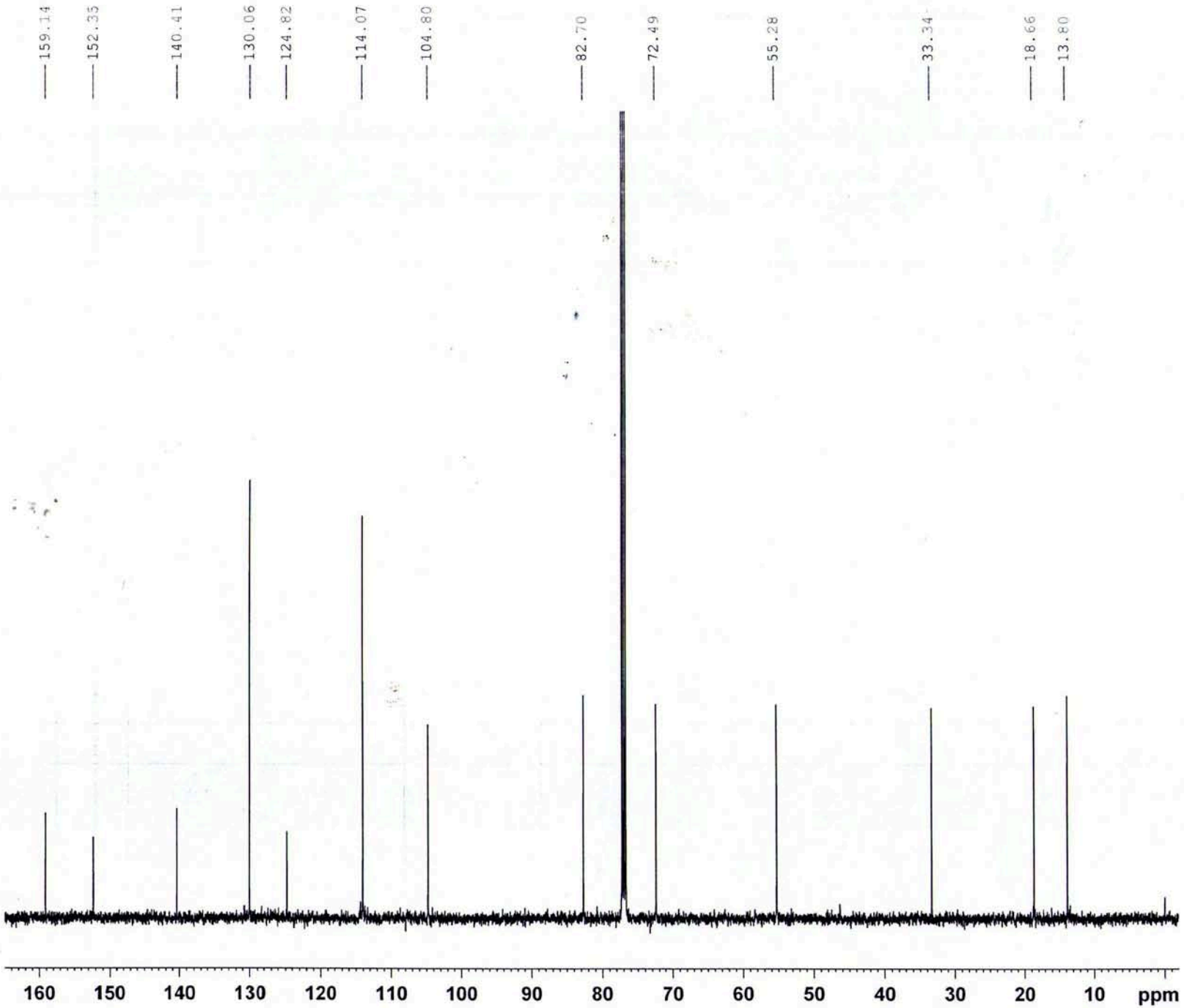
3g



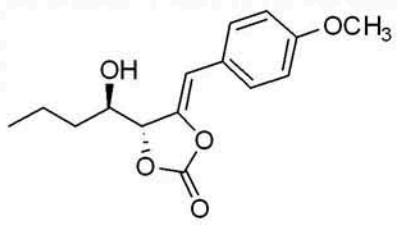
NAME	MB-317-1
EXPNO	1
PROCNO	1
Date_	20121010
Time	10.51
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	4826.255 Hz
FIDRES	0.147286 Hz
AQ	3.3948147 sec
RG	181
DW	103.600 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	9.35 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2621043 MHz
SI	32768
SF	500.2600145 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.00



3h

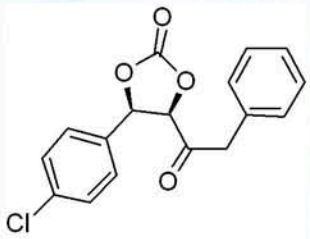


NAME	MB-317-1
EXPNO	2
PROCNO	1
Date_	20121010
Time	10.56
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	259
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	0.5505524 sec
RG	1030
DW	16.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.50 usec
PL1	3.00 dB
PL1W	32.22848892 W
SFO1	125.8043140 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.20 dB
PL12	18.40 dB
PL13	18.40 dB
PL2W	20.76952171 W
PL12W	0.39575511 W
PL13W	0.39575511 W
SFO2	500.2621041 MHz
SI	32768
SF	125.7904826 MHz
WDW	EM
SSB	0
LB	1.50 Hz
GB	0
PC	1.40

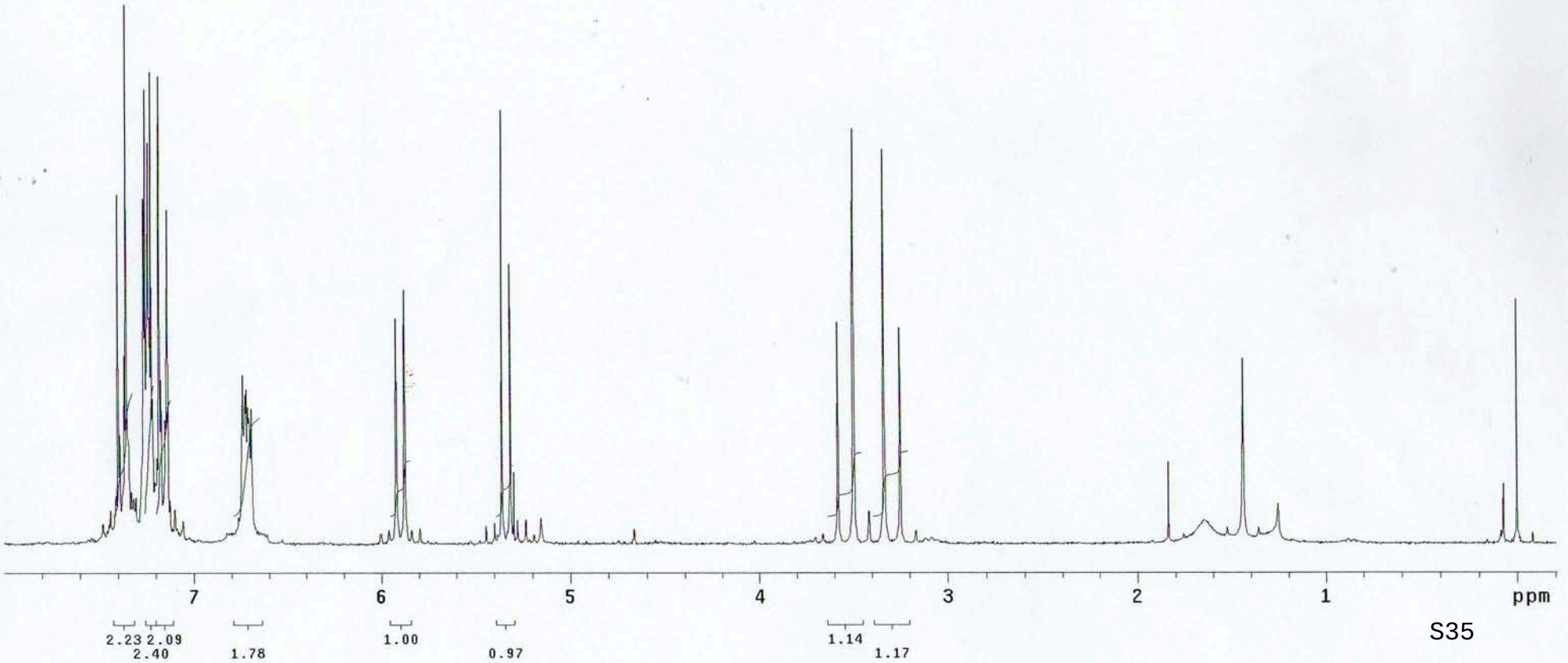


3h

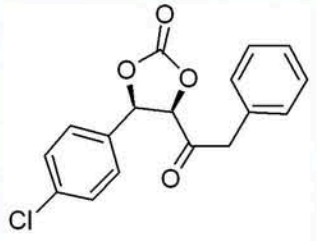
MB-SS-351
Solvent: cdcl3
Ambient temperature
File: hmbss351
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 90.0 degrees
Acq. time 1.437 sec
Width 4600.0 Hz
Arrayed repetitions
OBSERVE H1, 199.9710962 MHz
DATA PROCESSING
FT size 16384
Total time 2 minutes



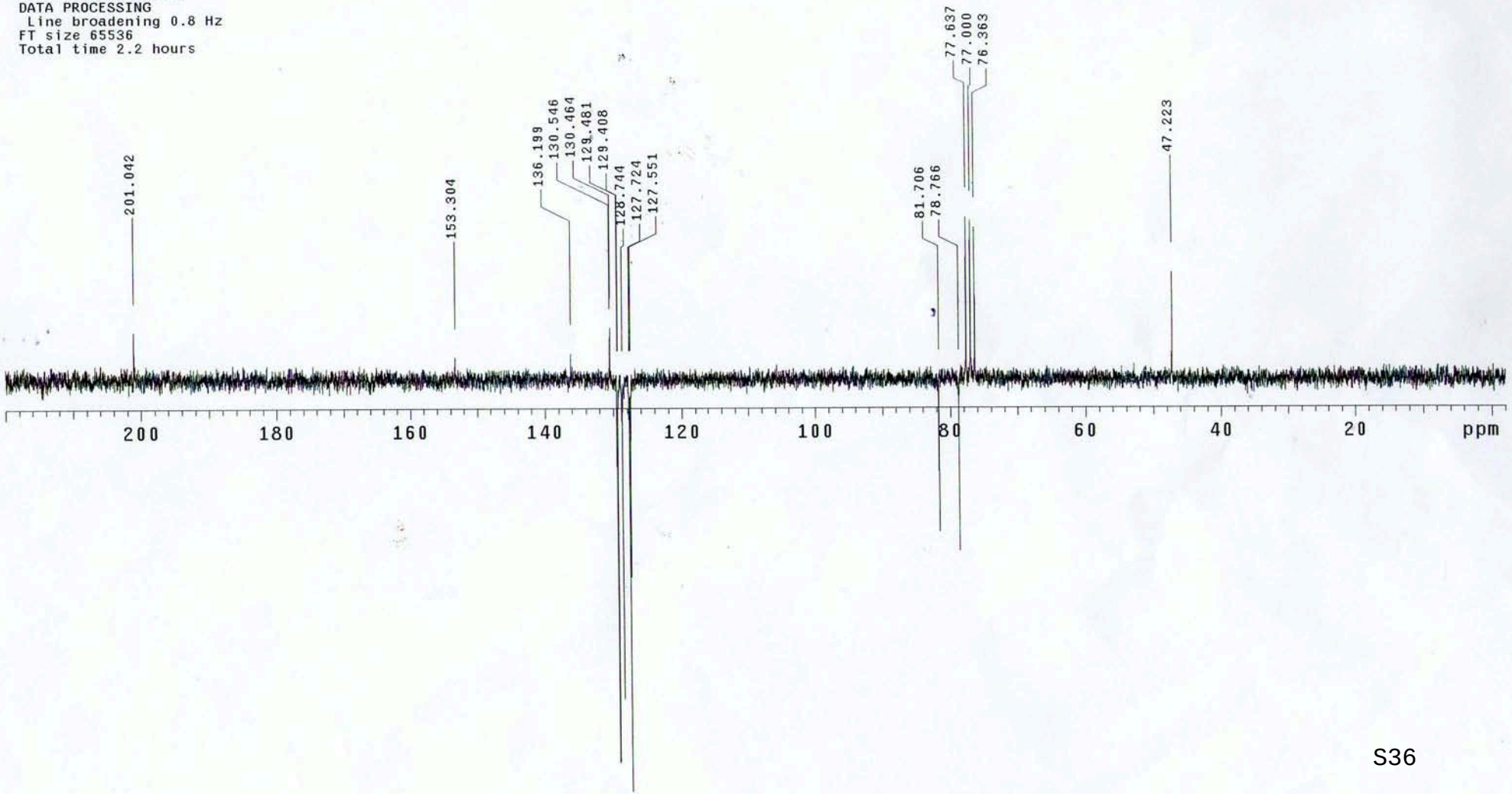
4a

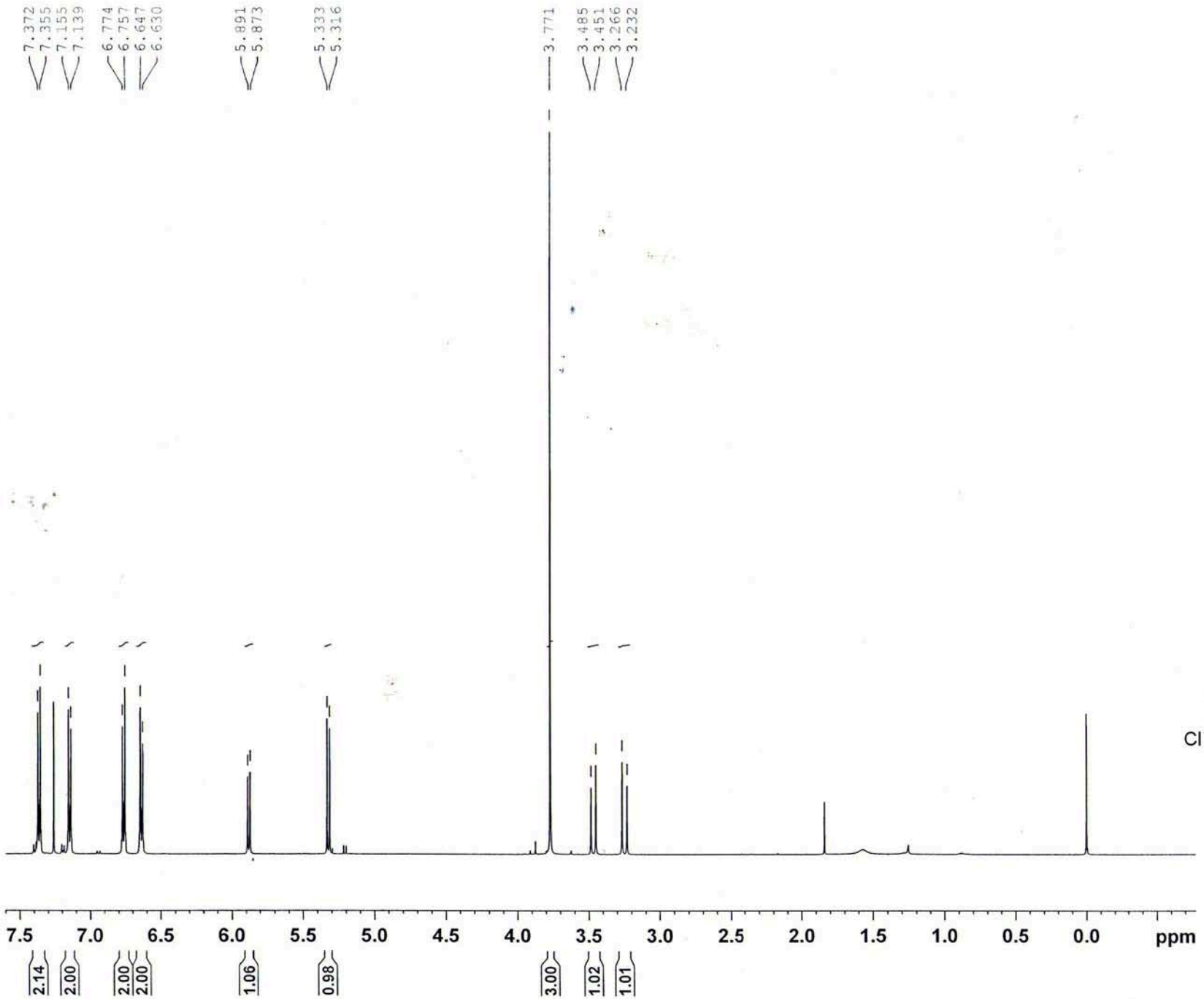


MB-SS-351
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE: apt
Relax. delay arrayed
1st pulse arrayed
2nd pulse 122.7 degrees
Acq. time 2.000 sec
Width 15000.0 Hz
Arrayed repetitions
OBSERVE C13, 50.2827785 MHz
DECOUPLE H1, 199.9712807 MHz
Power 0 dB
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 2.2 hours

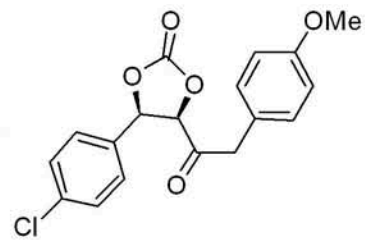


4a

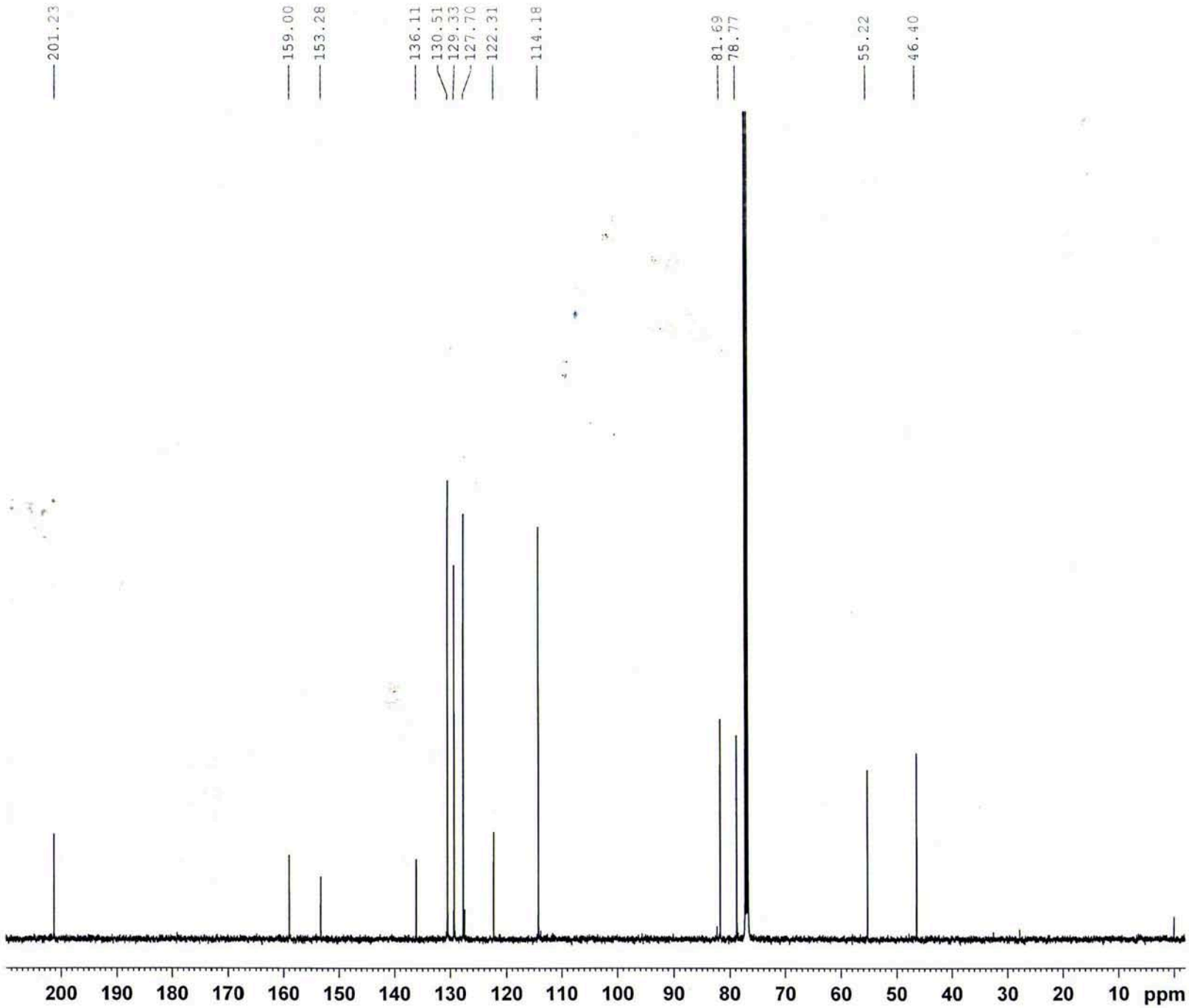




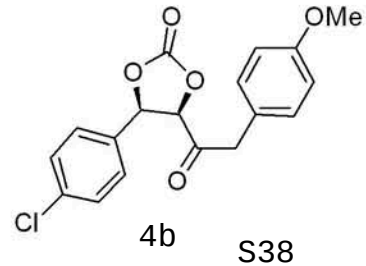
NAME	MB-289
EXPNO	1
PROCNO	1
Date_	20120817
Time	14.16
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	4629.629 Hz
FIDRES	0.141285 Hz
AQ	3.5389941 sec
RG	256
DW	108.000 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	9.35 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2619427 MHz
SI	32768
SF	500.2600148 MHz
WDW	EM
SSB	0
LB	0.20 Hz
GB	0
PC	1.00



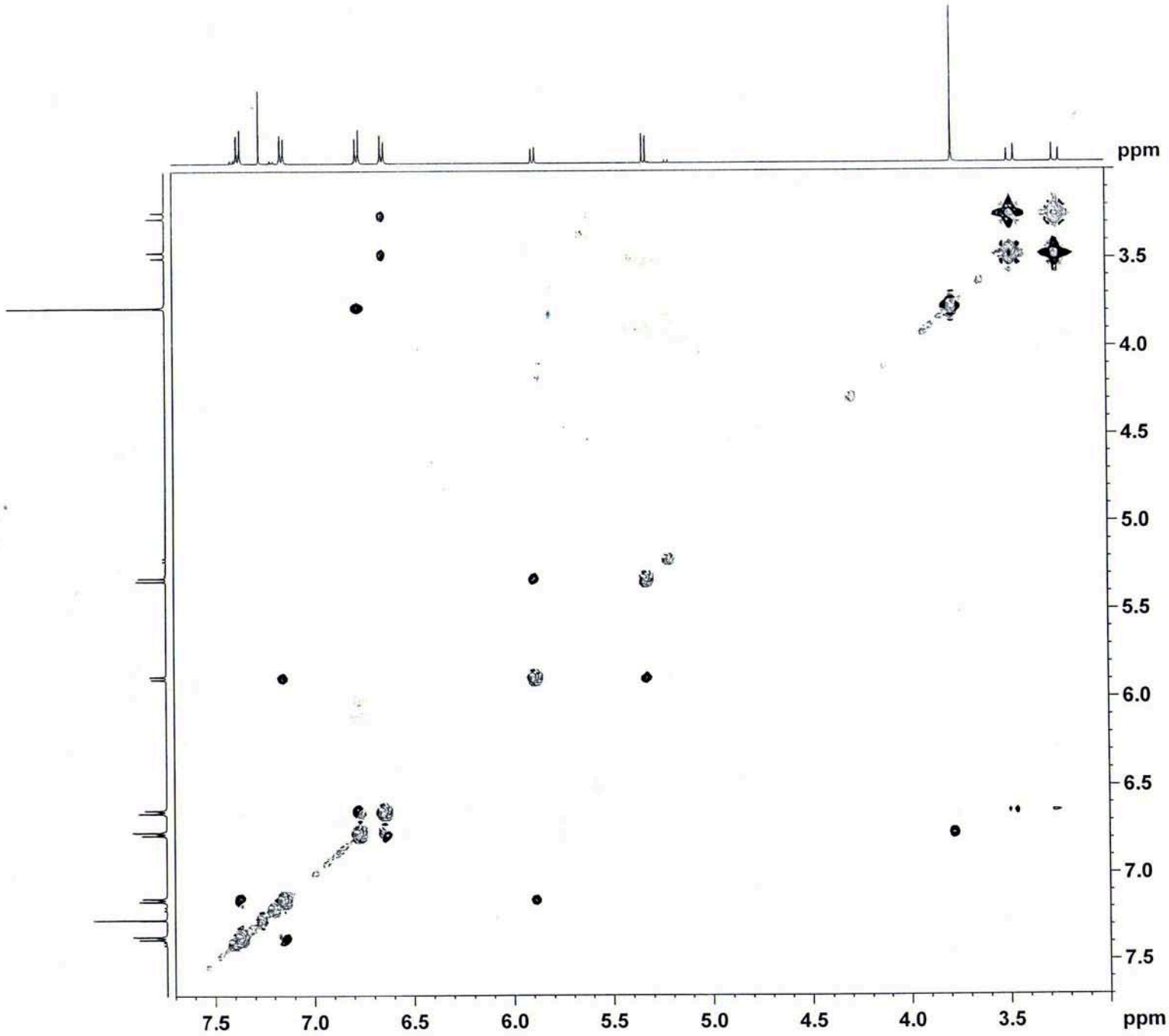
4b



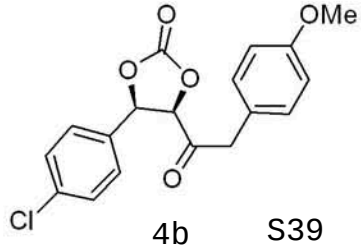
NAME	MB-289
EXPNO	2
PROCNO	1
Date_	20120817
Time	14.21
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	1651
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	0.5505524 sec
RG	1030
DW	16.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.50 usec
PL1	3.00 dB
PL1W	32.22848892 W
SFO1	125.8043140 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.20 dB
PL12	18.40 dB
PL13	18.40 dB
PL2W	20.76952171 W
PL12W	0.39575511 W
PL13W	0.39575511 W
SFO2	500.2619425 MHz
SI	32768
SF	125.7904814 MHz
WDW	EM
SSB	0
LB	1.50 Hz
GB	0
PC	1.40



4b

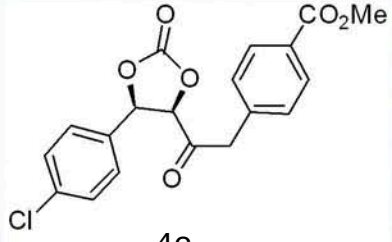


NAME	MB-286-3
EXPNO	4
PROCNO	1
Date_	20120814
Time	13.48
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	noesygpph
TD	1024
SOLVENT	CDCl3
NS	8
DS	16
SWH	4629.629 Hz
FIDRES	4.521122 Hz
AQ	0.1106420 sec
RG	287
DW	108.000 usec
DE	6.50 usec
TE	298.0 K
D0	0.00009610 sec
D1	2.00000000 sec
D8	1.00000000 sec
D16	0.00020000 sec
IN0	0.00021600 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	9.35 usec
P2	18.70 usec
PL1	0.00 dB
PL1W	27.37956238 W
SFO1	500.2620786 MHz
====== GRADIENT CHANNEL =====	
GPNAM1	SINE.100
GPZ1	40.00 %
P16	1000.00 usec
ND0	1
TD	256
SFO1	500.2621 MHz
FIDRES	18.084475 Hz
SW	9.254 ppm
FnMODE	States-TPPI
SI	512
SF	500.2600091 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	512
MC2	States-TPPI
SF	500.2600097 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

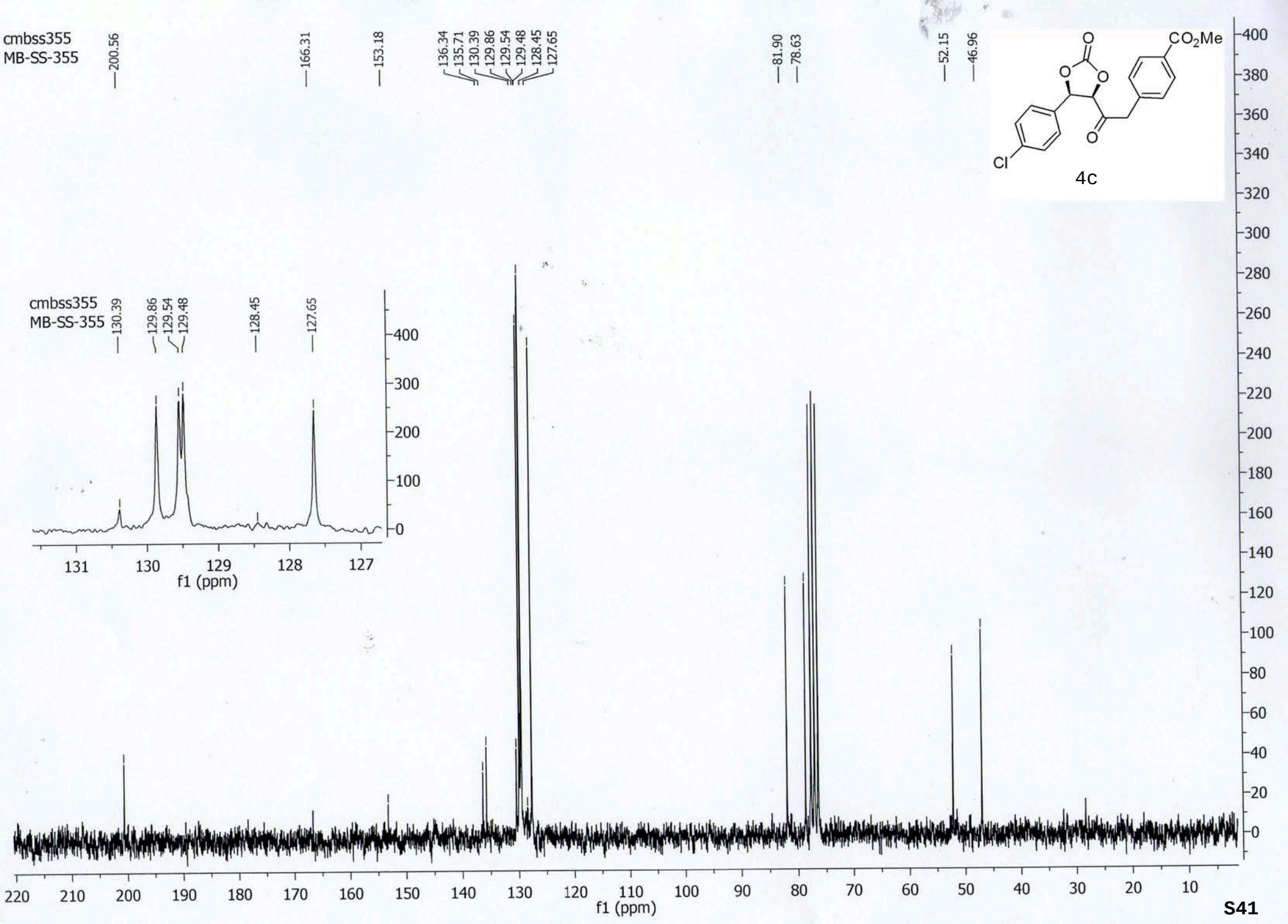


4b

MB-SS-355
Solvent: cdcl3
Ambient temperature
GEMINI-200 "nmr"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 90.0 degrees
Acq. time 1.437 sec
Width 4600.0 Hz
Arrayed repetitions
OBSERVE H1, 199.9710951 MHz
DATA PROCESSING
FT size 16384
Total time 2 minutes



4c


cmbss355
MB-SS-355
200.56
166.31
153.18
136.34
135.71
130.39
129.86
129.54
129.48
128.45
127.65
81.90
78.63
52.15
46.96
CO2Me
Cl
4c
cmbss355
MB-SS-355
130.39
129.86
129.54
129.48
128.45
127.65
f1 (ppm)
f1 (ppm)